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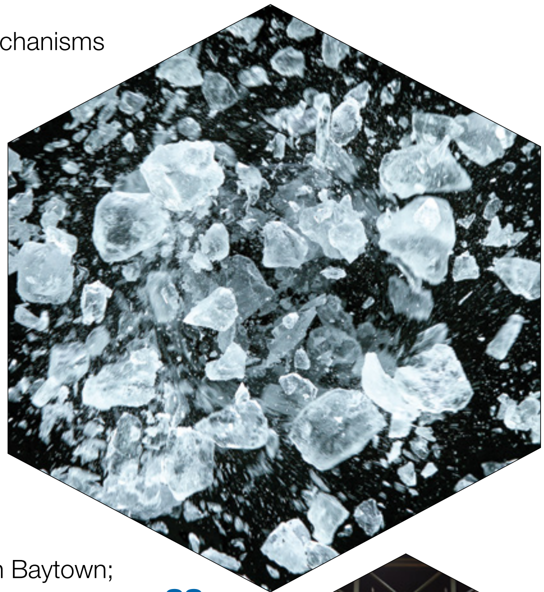
Cover Story



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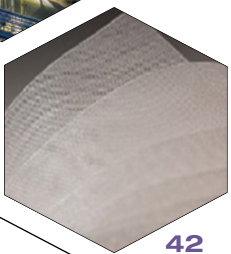

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Cover design: Tara Bekman

Editor's Page

The 46th Kirkpatrick Award winner

Chemical Engineering is pleased to announce that the winner of the 46th Kirkpatrick Chemical Engineering Achievement Award is Haldor Topsoe A/S (Lyngby, Denmark), for its hydrotreating catalyst TK-6001 HySwell. We congratulate Haldor Topsoe for this winning achievement.

Halsor Topsoe's TK-6001 HySwell is an alumina-supported NiMo hydrotreating catalyst. NiMo catalysts are in demand for ultra-low-sulfur diesel and hydrocracker pretreatment. Until now, unsupported catalysts have been the only type that could meet the high activity needed for these applications. Topsoe has developed catalyst preparation techniques to produce a stable, supported catalyst that can be regenerated. More about this winning technology will be included in *Chemical Engineering*'s January 2022 issue.

We also congratulate the four honoree finalists for the 2021 Kirkpatrick Award: BQE Water; Dow Industrial Intermediates & Infrastructure; Dow Deutschland; and Sapphire Technologies on their innovative technologies, which were outlined on this page in our July issue.

The award

The 2021 Kirkpatrick Chemical Engineering Achievement Award recognizes and honors a highly noteworthy chemical-engineering technology commercialized in 2019 or 2020. The award is given once every two years and was initiated in 1933, when *Chemical Engineering* was known as *Chemical and Metallurgical Engineering*. Formerly known as the Award for Chemical Engineering Achievement, it was renamed the Kirkpatrick Award in 1959 in honor of Sidney Kirkpatrick, who served as the editorial director of *Chemical Engineering* and *Chemical Week* (1950–1959). Kirkpatrick was also a former president of the American Institute of Chemical Engineers (AIChE) and of the American Electrochemical Society.

Haldor Topsoe joins a long list of winning achievements that include: LanzaTech for Emissions-to-Ethanol Fermentation Technology (2019); CB&I and Albemarle Corp. for the AlkyClean process (2017); Dow Performance Plastics for Intune Olefin Block Copolymers (2015); and Genomatica for its process to produce bio-based butanediol (2013). The full list of past winners can be found at www.chemengonline.com/kirkpatrick-award.

The selection process

The winner of the award is selected through a process whereby current chairs of chemical engineering departments at U.S. and E.U. universities vote to select finalists from all of the qualified entries, and then a smaller group of professors select the final winner. We thank this year's judges in that final selection process for taking the time to carefully review the finalists' submissions and contributing to honoring their achievements: Jean-Claude Charpentier, Université de Lorraine École Nationale Supérieure des Industries Chimiques, France; Abhaya K. Datye, University of New Mexico; Mario Richard Eden, Auburn University, Alabama; Mark Swihart, University at Buffalo, New York; Thomas Turek, TU Clausthal, Germany; and Michael S. Wong, Rice University, Texas. ■

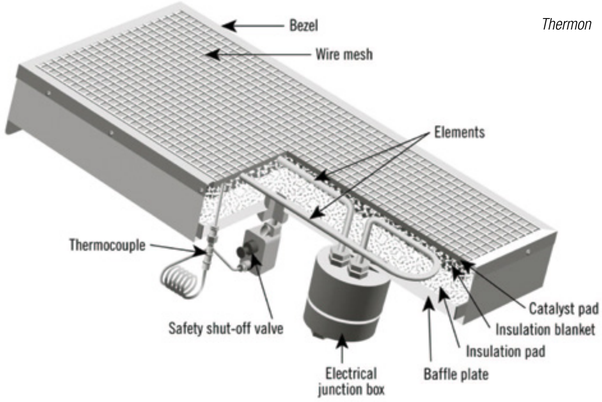
Dorothy Lozowski, Editorial Director

Combustion-free abatement of fugitive methane emissions

Curbing emissions of methane gas is a major concern for many industrial sectors. Typical emissions-abatement methods involve combustion, but a new flameless technology designed by Thermon, Inc. (Austin, Tex.; www.thermon.com) employs a proprietary explosion-proof catalytic pad in place of a combustion reaction, making it safer to use while also creating fewer emissions.

Thermon's EnviroDyne Methane Destruction Unit (MDU; diagram) can convert over 90% of fugitive methane emissions into carbon dioxide and water vapor using principles similar to the catalytic converters found in vehicles. "Since the conversion process takes place at a lower temperature than conventional flaring, it can be used in designated hazardous locations where potentially explosive gases are present," explains Thermon product manager, Dave TenEycke. The unit is certified to meet requirements for use in Class I, Div. 1 & 2 hazardous locations, making it much more versatile than flaring to convert fugitive emissions from a wide variety of pneumatic devices in locations where flames could create serious explosions. "We do not know of another certified product of this type designed for methane abatement," says TenEycke. Furthermore, he adds: "EnviroDyne's lower operating temperature also means that it does not generate harmful oxides of nitrogen (NOx) emissions, which do occur during flaring."

Another benefit of the MDU is that it has no moving parts and requires little human intervention during extended operation. "The unit requires only electricity, often via a battery, during initial startup and can run indefinitely once started, as long as clean fuel and adequate air supply are available," says TenEycke. The technology has been trialed at multiple sites and is currently available in several sizes and voltages.



Feed injector lowers energy consumption of FCC units

A newly launched feed injector for fluid catalytic cracking (FCC) units at petroleum refineries reduces the oil-side pressure drop and lowers the steam required, enabling facilities to achieve the same performance with lower energy consumption, according to developer Lummus Technology (Houston; www.lummustechnology.com). Known as the Micro-Jet Flex, the new feed injector is a next-generation version of the company's Micro-Jet product line. Lummus' development engineers have re-designed the injector's internals to maximize surface-area contact between the liquid oil feed and the circulating solid-catalyst particles. To achieve this, the injector reduces droplet sizes, narrows the droplet size distribution and optimizes the spray pattern into the catalyst stream.

"The reduced droplet size and narrow size distribution allowed by the new injector ultimately translates into less coke formation and more conversion of heavier feeds into olefins," says Rama Rao Marri, senior director of FCC technology at Lummus Technology.

The Micro-Jet Flex can be easily retrofitted into any existing FCC unit, and is a simple drop-in replacement for plants that are already using other Micro-Jet products, remarks Todd Vogt, vice president and managing director for refining and gas processing at Lummus. With the Micro-Jet Flex, "we've built on the same principles at play in previous injectors, but to a much greater effect," says Vogt. "The introduction of the Micro-Jet Flex is part of a wider effort by Lummus to support refineries seeking to increase production of petrochemical feedstocks, such as light olefins, in their FCC units."

The injector has been tested at Lummus' R&D facilities and is currently undergoing engineering design for the first installation in a commercial facility, notes Vogt.

Edited by:
Gerald Ondrey

ODH CATALYST

Clariant Catalyts (Munich; www.clariant.com) has teamed up with Linde Engineering (Pullach im Isartal, both Germany; www.linde-engineering.com) to develop new catalysts for the oxidative dehydrogenation of ethane (ODH-E), a low-emissions catalytic technology for the production of ethylene. The new catalyst is said to be a step change in selectivity and productivity, now making ethylene production via ODH-E commercially feasible.

Clariant's ODH catalysts are available exclusively for Linde Engineering's EDHOX catalytic on-purpose ethylene technology. In contrast to conventional steam cracking, which operates at process temperatures up to 900°C, EDHOX operates at moderate temperatures (below 400°C), enabling comparatively low CO_2 emissions.

The new catalysts by Clariant are the first to offer high selectivity for ethylene and acetic acid (up to 93%) for ODH-E, while also demonstrating outstanding productivity at such conditions, minimizing the formation of byproducts.

"By working with Clariant Catalysts, a more sustainable, alternative path to ethylene is not only a vision, but also becoming a reality," says Reinhart Vogel, vice president Petrochemical Plants at Linde Engineering. "EDHOX technology has now been successfully validated in a full-scale demonstration plant for commercial use."

TUNGSTEN

H.C. Starck Tungsten Powders GmbH (Goslar, Germany; www.hcstarck.com), a wholly

(Continues on p. 6)

owned subsidiary of Vietnam listed Masan High Tech Materials, has developed a new process for high-pressure reverse osmosis in the production of tungsten chemicals that significantly reduces energy consumption and CO_2 emissions compared to conventional processes. To achieve this, the Technology and Innovation department cooperated closely with process specialist OSMO Membrane Systems GmbH (Korntal-Münchingen, Germany; www.osmo-membrane.de). H.C. Starck is already using the process at its Goslar site for the concentration of ammonium metatungstate (AMW) solutions on an industrial scale.

In the production of AMW, a very dilute solution is first obtained from ammonium paratungstate (APW), which then has to be highly concentrated. This is traditionally done by evaporation, which is energy intensive. With the new patent-pending process, the solution is pressed under high pressure through a semi-permeable membrane. The membrane-based approach reduces energy consumption by more than 95% at a production volume of around 1,000 ton/yr of AMW, says the company.

CO_2-NEUTRAL CH_3OH

Haldor Topsoe A/S (Lyngby, Denmark; www.topsoe.com) has started up a demonstration plant for the production of sustainable methanol from biogas. The demonstration plant, which is part of a project supported by the EUDP Energy Technology Development and Demonstration Program and is developed together with Aarhus University, aims to validate Topsoe's electrified steam-methane-reforming (eSMR) technology for cost-competitive production of sustainable methanol from biogas as well as other sustainable products.

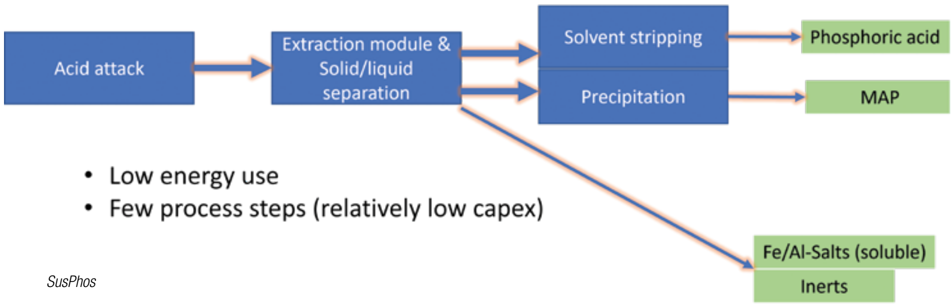
As with conventional SMR, eSMR reforms methane into synthesis gas (syngas), which can then be used for methanol synthesis (or other chemicals). However, instead of heating the catalyst-packed reformer tubes indirectly by the combustion of fuels, eSMR uses smaller-diameter tubes, which

(Continues on p. 8)

Recovering phosphoric acid from sewage-sludge incineration ash

This month, SusPhos B.V. (Amsterdam, the Netherlands; www.susphos.com) is increasing the capacity ten-fold of its 25-kg/d pilot plant in Leeuwarden, the Netherlands. The pilot plant, which began operating in January, is being used to develop a process that recovers phosphorus from sewage-sludge incinerator ash, and produces merchant-grade phosphoric acid (MGA), and mono- and diammonium phosphate (MAP, DAP), while recovering other metal salts. "Our main goal is to recover phosphorus without generating any waste — to recycle everything," says chief technology officer Willem Schipper.

In the process (diagram), incinerator ash is first treated with acid to generate phosphoric acid. In a second step, a proprietary organic solvent is used to selectively extract this H_3PO_4, which is then stripped of solvent to produce 75–80 wt.% H_3PO_4 with very low metals content. Meanwhile, the remaining solids are treated to recover iron and aluminum salts as side products, leaving behind an insoluble, inert-mineral stream in which heavy metals are fixed. The process is flexible, so that the phosphate can alternatively be recovered as a MAP or DAP flame retardant and fertilizer in a simple add-on step.



SusPhos

The SusPhos process uses an order of magnitude less water and energy, when compared to alternative phosphorus-recovery methods that require energy-intensive water evaporation to concentrate the acid or salt solutions, says Schipper.

The company is simultaneously carrying out the basic engineering and design for a full-scale plant that can treat 50,000 ton/yr of incinerator ash. "We are ready to offer a 'ripe' technology for when it is needed," says Schipper, who expects a first plant within the next two years. In Germany, for example, phosphorus-recovery from sewage will be required in 2029, he says.

Self-healing hydrophobic coatings enhance heat transfer in steam condensers

Ultrathin (less than 100 nm) hydrophobic coatings on alloys and other engineering materials could enhance heat and mass transfer in a range of processing applications, but achieving lasting durability for such thin coatings in real-world settings has been a major ongoing challenge. Now, researchers at the University of Illinois (Champaign, Ill.; www.illinois.edu) have developed a coating material that can repair itself after scratching, cutting and other damage, extending its durability even at nanoscale thicknesses.

The current research project, led by University of Illinois engineering professor Nenad Miljkovic and published in *Nature Communications*, focused on using the coatings to boost the efficiency of condensers in power-plant steam systems. Hydrophobic coatings render the metals more water-resistant and efficient at forming water droplets, which enhances heat transfer, the team notes. In steam power plants, thin coatings can break down quickly. Thicker coatings can be more durable, but they reduce heat transfer and erode the associated benefit of the coating.

To avoid this tradeoff, the researchers designed and synthesized a vitrimer thin film with polydimethylsiloxane network strands and dynamic boronic ester crosslinks. Named dyn-PDMS, the coating material takes advantage of the inherent hydrophobic nature of silicones, and provides a mechanism for self-healing, due to the dynamic exchange of bonds in their network strands, the researchers say. Vitrimers refer to a class of materials with covalent bond networks that can undergo bond exchange.

The film "maintains excellent hydrophobicity and optical transparency after scratching, cutting, and indenting," the researchers say. In addition to enhanced heat transfer, the coating material could have a range of other potential applications, such as self-cleaning, anti-icing, anti-fogging, anti-bacterial or anti-fouling coatings.

The dyn-PDMS can be easily dip-coated onto surfaces — including silicon, aluminum, copper and steel — in nanoscale layers.

Blast-furnace technology aims for carbon neutrality

Primetals Technologies, Ltd. (London, U.K.; www.primetals.com) and thyssenkrupp AG (Essen, Germany; www.thyssenkrupp.com) have signed an exclusive, worldwide cooperation agreement that sees both parties commit to bringing the Sequence Impulse Process (SIP) technology to the market. The SIP technology has been developed by thyssenkrupp AT.PRO tec GmbH, a company of thyssenkrupp Materials Services GmbH (www.thyssenkrupp-materials-services.com), and is well established for use in foundry cupola furnaces. The underlying principles of this proven technology have been adapted and specifically tailored to the blast-furnace process. To prove the technology, thyssenkrupp Steel Europe has installed the first ever full system on their blast furnace #1 at Schwelgern, Duisburg in Germany (photo).


Thyssenkrupp

Blast-furnace operators can conventionally lower operating costs through injection of alternative fuels with the hot-air blast (most commonly pulverized coal) in order to reduce the required coke rate in the burden. Additional O_2 enrichment is required to promote combustion for the added fuel. While benefiting the economic aspects for the furnace, this also results in a number of process and operational challenges. Combustion of the fuel localized in the raceway entry leads to higher heat loads on the shell, and uncombusted material (char) can accumulate in the burden. This impedes gas flow and drainage, ultimately reducing the furnace performance efficiency.

The patented SIP technology acts to repurpose the enrichment O_2, and deliver a series of controlled, high-energy pulses, increasing local concentration in the raceway. The resulting action is improved combustion and conversion of fuel, leading to better gas distribution and drainage potential.

Additionally, through higher penetration of the oxygen, the heat load generated is moved more to the central zone of the furnace. The small coke proportion in the burden can be increased without loss of permeability or increased overall pressure drop. These conditions permit an increase in the replacement of coke with injectant and higher production potential while avoiding deterioration of the process conditions.

"By optimizing the consumption of the reducing agents coke and coal, we have a major lever for efficiency improvements, cost and CO_2 savings," says Jörg Glebe, managing director of thyssenkrupp AT.PRO tec.

have a catalyst coated on the inner walls, that are directly heated electrically. The company has achieved 10-times higher catalyst efficiency with this technology. As a result, the size of the reformer can be reduced by a factor of 100.

The demonstration plant — located at Aarhus University's research facility in Foulum, Denmark — will have a capacity of 10,0000 L/yr of CO_2-neutral methanol from biogas and green power and is scheduled to be fully operational by the beginning of 2022.

Topsoe is leading the project, with partners Aarhus University, Sintex, Blue World Technology, Technical University of Denmark, Energinet, Aalborg University, and PlanEnergi.

ETHANOL SENSOR

Researchers at the Karlsruhe Institute of Technology (KIT; www.kit.edu) and the Technical University of Darmstadt (both Germany; www.tu-darmstadt.de) have developed a new sensor for gas molecules that responds selectively to ethanol, but not other alcohols or humidity. Described in a recent issue of *Advanced Materials*, this new class of sensor is based on the combination of sensitive graphene transistors with a customized metal-organic framework (MOF) grown on the surface. MOFs consist of metallic nodes and organic molecules as connecting rods. By choosing various combinations, these highly porous crystalline materials can be tailored to different applications to reach a selective absorption capacity for certain molecules, for instance. The researchers from Karlsruhe and Darmstadt presented a selective sensor platform by growing a surface-mounted metal-organic framework (SURMOF) directly on a graphene field-effect transistor (GFET). Such a component profits from the high sensitivity and simple read-out of a GFET, as well as from the high selectivity of a SURMOF.

BIO-COMPOSITE

Last month, Lanxess AG (Cologne, Germany; www.lanxess) introduced a new variant of the composite Tepex that is 100% based on biological raw materials flax and polylactic acid. The extremely strong material is suitable for use in sports articles, in the production of automotive interior parts and in electronics for case components. Lanxess produces the composite at its site in Brilon, Germany. Tepex can be completely recycled. ❐

Paving the way for emissions-free renewable fuels

UOP, LLC, a Honeywell company (Des Plaines, Ill.; www.uop.com) and Wood plc (Aberdeen, Scotland; www.woodplc.com) are now licensing a combined technology suite that brings together UOP's Ecofining renewable-fuels process with Wood's Terrace Wall hydrogen-reforming furnace, with the aim of substantially reducing greenhouse-gas emissions in the production of sustainable fuels. The main synergy between the technologies comes from the efficient utilization of Ecofning's byproducts.

"We're taking some of the secondary products from the Ecofining technology, which are primarily renewable naphtha and renewable liquefied petroleum gasoline (LPG), and instead of using those byproducts as fuel blendstocks, we're sending them to Wood's hydrogen unit as fuel to be converted into a purified, renewable-hydrogen feedstream that is recycled back as feed to the Ecofining unit," explains Dan Szeezil, renewable fuel leader at Honeywell. Because the hydrogen that is being used for the Ecofining renewable diesel process is actually produced from the same renewable feed — such as used cooking oil or animal fats — that is used to produce renewable-diesel and jet fuel, the carbon intensity of the process is significantly reduced, adds Szeezil. "Furthermore, as part of this overall combined suite, UOP offers CCS [carbon capture and storage] technology for the CO_2 emissions that come from the hydrogen unit," says Szeezil.

The first commercial demonstration of this integrated process will take place at ECB Group's renewable-fuels project in Villeta, Paraguay, which is slated to produce up to 20,000 barrels per day (bbl/d) of renewable diesel and jet fuel.

Improved process for recycling used motor oil produces higher-grade base oils

Front-end engineering design (FEED) is underway for a facility designed to de-contaminate and upgrade used motor oil from passenger vehicles into a higher-value product mix of base oils than is currently possible with existing motor-oil recycling processes, and in a way that lowers capital and operating costs compared to current processes. The project is being undertaken by ReGen III Corp. (Vancouver, B.C.; www.regeniii.com), a technology developer focused on re-refining used motor oils, and is supported by engineering and modular construction firm Koch Modular Process Systems LLC (KMPS; Paramus, N.J.; www.kochmodular.com), which developed extraction and distillation technologies used in the process.

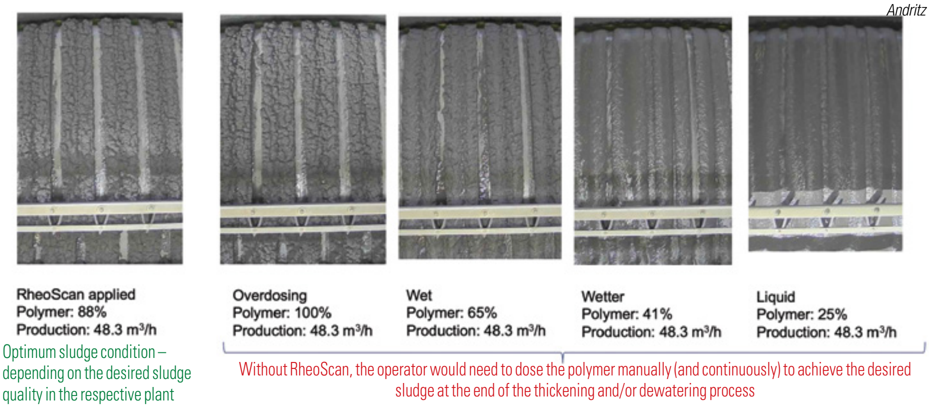
Initially, collected used motor oil enters a contamination separation unit (CSU), where low- and high-boiling compounds are removed to achieve a desired boiling-point cut. "This step removes some impurities and color, and allows for variability in feeds because it refines any feedstock into a similar boiling point range," explains Mark Redcliffe, executive vice president at ReGen III. The next stage involves a combination of liquid-liquid extraction and distillation. "The extraction step principally removes color bodies and improves certain physical properties, such as viscosity," says Wendy Parker, vice president of process engineering and pilot testing at KMPS. The distillation focuses on removing and recovering the solvent from the extraction-product streams (extract and raffinate). From this step, a high-quality Group III base oil is produced (up to 65 vol.%) that does not require further processing.

"Competitive processes are not generating Group III base oils, a higher-value product than the Group I and II oils typically generated in used motor oil recycling," says Redcliffe. Group II base oils are also produced (up to 25%) and continue to hydrotreating in the third stage. While alternative processes hydrotreat all incoming material, the ReGen III process requires hydrotreating for only about 30 vol.% of what comes from the CSU, explains Redcliffe, so the new process employs a smaller hydrotreating unit, resulting in lower capital costs. And since less consumable material is needed, operating costs are also lower, he adds.

Additionally, the ReGen III process utilizes liquid hydrogen, which allows for greater saturation of the lubricant molecules and results in a higher-grade Group II-plus base oil product, Redcliffe notes.

FEED for the project is scheduled to conclude in Q1 2022, and commissioning the finished re-refinery is currently planned for the second half of 2023, project leaders say.

AI-supported polymer-dosage system slashes operating costs



Andritz

Last month, Andritz AG (Graz, Austria; www.andritz.com) introduced its next-generation of Metris addIQ RheoScan, the world's first patented, optical-measurement system for automatic polymer dosage in belt presses and gravity belt tables. The new generation uses artificial intelligence (AI) to optimize polymer dosing. Savings in polymer lead to operating expenditure (opex) reduction by up to 40% for sewage sludge, coal sludge or paper sludge dewatering, the company says. Using the "cutting-edge" technologies also help to maintain optimum cake dryness and to identify blinded belts, which in turn leads to smoother operations. Installations in wastewater treatment plants with digestion processes have seen the additional benefit of higher biogas yields. The new squeeze-control option enables the system to operate at the capacity limits of the machine 24/7, effectively eliminating downtime.

With Metris addIQ RheoScan, an imaging system takes pictures of the sludge surface. Each picture is divided into multiple sub-images that are classified by a previously trained convolutional neural network (CNN) regarding its particular flocculation condition, which are represented by texture and color of the sludge (see example photos). The total degree of flocculation is then calculated over all images (0% wet, 100% dry) and compared to a setpoint. A controller continuously adjusts the polymer flow to keep the degree of flocculation at the setpoint.

Metris addIQ RheoScan measures the actual sludge conditions throughout the thickening and dewatering stages in real time, without supervision, and can be applied to any belt press or gravity belt table in all types of municipal and industrial sludge-processing facilities. Users who have installed a Metris addIQ RheoScan generally recouped their investment in less than a year, says the company.

Business News

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Plant Watch

ExxonMobil to build large-scale advanced recycling facility in Baytown

October 11, 2021 — ExxonMobil Corp. (Irving, Tex.; www.exxonmobil.com) plans to build its first large-scale, advanced recycling facility for plastic waste in Baytown, Tex. Expected to start operations by year-end 2022, the plant will have an initial planned capacity to recycle 30,000 metric tons per year (m.t./yr) of plastic waste. The company also plans to add up to 500,000 m.t./yr of advanced recycling capacity at other sites by year-end 2026.

LG Chem to construct new ABS plant in Ohio

October 11, 2021 — LG Chem Ltd. (Seoul, South Korea; www.lgchem.com) will invest around 60 billion won (around $50 million) to construct an acrylonitrile butadiene styrene (ABS) plant at the company's Ohio Tech Center. The new plant is slated for startup by 2023, and will have a production capacity of 30,000 m.t/yr of ABS.

Arkema to build a bio-based polyamide 11 plant in China

October 11, 2021 — Arkema S.A. (Colombes, France; www.arkema.com) is constructing a bio-based polyamide 11 powders plant at its Changshu site in China. The new plant is scheduled to come onstream in early 2023. Arkema currently produces bio-based polyamide 11 powders in France, and construction is underway for an additional plant in Singapore.

Air Products starts up La Porte liquid hydrogen plant

October 8, 2021 — Air Products (Lehigh Valley, Pa.; www.airproducts.com) started up a new 30-ton/d liquid hydrogen plant at its La Porte, Tex. facility. The plant will draw its hydrogen to be liquefied from Air Products' existing Gulf Coast hydrogen pipeline, the world's largest hydrogen plant and pipeline system.

Sabic starts up PP compounding line in Belgium

October 7, 2021 — Sabic (Riyadh, Saudi Arabia; www.sabic.com) announced the startup of a new polypropylene (PP) compounding line in Genk, Belgium. The new line is an addition to the company's existing production capacity for PP compounds at the Genk site and will use raw materials from Sabic's PP plants in Germany and the Netherlands.

BP to double renewable-diesel production capacity at Cherry Point Refinery

October 5, 2021 — BP plc (London; www.bp.com) announced a $45-million investment that will more than double the renewable-diesel production capacity of the BP Cherry Point Refinery in Washington state to an estimated 2.6 million bbl/yr. Renewable diesel is manufactured from biomass-based feedstocks, such as vegetable oils and rendered animal fats.

TotalEnergies expands recycled polypropylene production capacity

October 4, 2021 — TotalEnergies (Paris; www.totalenergies.com) has expanded the Synova recycled PP production site in Normandy, France. With the installation of two new production lines, the Synova site will produce almost 45,000 m.t./yr of recycled PP using mechanical recycling methods.

Mitsubishi Gas Chemical to expand MXDA capacity in Europe

September 30, 2021 , Mitsubishi Gas Chemical Co. (MGC; Tokyo; www.mgc.co.jp) is building a new plant in Rotterdam, the Netherlands to produce *meta*-xylenediamine (MXDA). The new plant is scheduled to start operation in mid-2024 and is slated to produce around 25,000 m.t./yr of MXDA.

Nouryon starts production of TBHP and TBA at new plant in Ningbo

September 29, 2021 — Nouryon (Amsterdam, the Netherlands; www.nouryon.com) has started production at a new manufacturing facility located at its site in Ningbo, China. The facility, which began development in 2020, has a production capacity of 35,000 m.t./yr to produce two key intermediates — *tert*-butyl hydroperoxide (TBHP) and *tert*-butyl alcohol (TBA) — that are essential in the production of polymers and composites.

Merck opens new pigments production unit in Germany

September 21, 2021 — Merck KGaA (Darmstadt, Germany; www.merckgroup.com) has opened a second production line for silica-based effect pigments in Gernsheim, Germany. With this €28-million investment, Merck will significantly increase production capacities for silicon dioxide flakes.

Huntsman announces specialty amines expansion project

September 20, 2021 — The Performance Products division of Huntsman Corp. (The Woodlands, Tex.; www.huntsman.com) has completed the initial phases of a project to expand and widen its portfolio of specialty amines, including quaternary amine alternatives to tetramethylammonium hydroxide (TMAH) and related products. Located at Huntsman's facility in Conroe, Texas, the new production plant is expected to be completed and operational in 2023.

Mergers & Acquisitions

Emerson to merge software businesses with AspenTech

October 11, 2021 — Emerson (St. Louis, Mo.; www.emerson.com) and AspenTech, Inc. (Bedford, Mass.; www.aspentech.com) have agreed to merge Emerson's industrial software businesses — OSI Inc. and the Geological Simulation Software business — with AspenTech to create a new diversified software organization (new AspenTech). Emerson will also contribute $6 billion to new AspenTech in exchange for a 55% stake in new AspenTech. The agreement values the transaction at approximately $11 billion.

Neste to sell base oils business to Chevron

October 5, 2021 — Neste Corp. (Espoo, Finland; www.neste.com) has signed an agreement to sell its existing base oils business to Chevron Corp. (San Ramon, Calif.; www.chevron.com). The agreement covers a combination of share and asset deals forming Neste's entire global base oils business. As part of the divestment, the parties have also agreed on a longterm offtake for Neste's base oils supply from Porvoo, Finland. Neste also will exit its base oils JV with Bahrain Petroleum Co. and Nogaholding.

Eastman completes acquisition of Matrix Films and PremiumShield

October 4, 2021 — Eastman Chemical Co. (Kingsport, Tenn.; www.eastman.com) has completed the acquisition of the business and assets of Matrix Films, LLC and its U.K. affiliate, PremiumShield Ltd. The newly acquired businesses joined Eastman's Performance Films business of the company's Advanced Materials segment.

Albemarle to acquire lithium conversion company in China

September 30, 2021 — Albemarle Corp. (Charlotte, N.C.; www.albemarle.com) will acquire Guangxi Tianyuan New Energy Materials Co. (Tianyuan), a lithium converter located in Guangxi, China, for approximately $200 million. Tianyuan's operations include a recently constructed lithium-processing plant strategically positioned near the Port of Qinzhou in Guangxi. The plant is designed to have a conversion capacity of up to 25,000 m.t./yr. It currently is in the commissioning stage and is expected to begin commercial production in the first half of 2022.

Cargill to acquire Arkema's epoxides business

September 27, 2021 — Cargill, Inc. (Minneapolis, Minn.; www.cargill.com) has entered into an agreement to acquire Arkema's epoxides business, which includes a production facility located in Blooming Prairie, Minn. After closing, the investment will give Cargill end-to-end production capabilities in bio-based plasticizers and polyols. The offer values this business at $38.8 million.

Kraton to be acquired by Korean firm DL Chemical

September 27, 2021 — Kraton Corp. (Houston; www.kraton.com) entered into a definitive merger agreement under which DL Chemical Co. (Seoul, South Korea; www.dlchemical.co.kr) will acquire 100% of Kraton in an all-cash transaction implying an enterprise value of approximately $2.5 billion. ■

Mary Page Bailey

A Holistic Approach to Water Management

Advanced equipment, new chemistries and digital technologies help manage water use to provide efficiency, sustainability and savings

Water, the "common currency" across all chemical process industries (CPI) sectors, is an integral part of every process — from upstream, where it is treated and used for utility and process purposes, to downstream, where it must again be treated to meet environmental regulations before discharge. However, as demand for water increases, water stress and scarcity are creating a situation where water is a costly and valuable resource. "Chemical processors have transitioned from considering water to be secondary to their production to realizing that water management and treatment is an integral part of the process, which means they are more focused on improving the efficiency and sustainability of this critical fluid," says Subir Bhattacharjee, founder and CEO of IntelliFlux Controls (Irvine, Calif.; www.ifctrl.com).

This new focus has led to the use of more sustainable water practices, and these practices do not stop at treating wastewater to comply with environmental regulations, says Charlotte Fischer, customer success manager with TrendMiner (Houston; www.trendminer.com). "Processors are focused on minimizing the amount of water used and the amount of wastewater produced, as well as changing the entire production process to include recycling or reusing water in a circular economy."

Today's version of a circular economy involves a regenerative system in which resource usage, waste production, emissions and energy waste are reduced by slowing, reducing and closing material and energy cycles. "In this circular economy, water, energy consumption and carbon footprint are often harnessed together, since water reuse and treatment are intrinsically energy intensive due to the need to move large streams of water," notes Fischer (Figure 1).

While mechanical and other treatment methods are improving radically to meet today's water challenges, digitalization and data analytics are also being employed to intensify the efficiency of water management technologies (Figure 2). "A digital and software-based upgrade often shows processors how to enhance the process intensity or process efficiency to achieve savings in operating expenses while still improving the sustainability of the current process," says Bhattacharjee.

Peter Macios, executive product manager with SUEZ Water Technologies & Solutions (Trevose, Pa.; www.suezwatertechnologies.com) agrees: "Sustainability and operations don't have to be exclusive. As operations evolve and chemical processors are reusing more water, it is a mix of equipment, new treatments and digital solutions that can help them achieve both sustainability and operational goals."

Equipment advances

Because sustainability includes reduced resource and energy consumption, along with increased efficiency, in a way that complies with regulatory frameworks, there are a myriad of technologies available to meet the needs of many situations, says Fischer. These can include zero liquid discharge (ZLD) technologies, advanced biological processes that use microorganisms to degrade organic matter, advanced oxidation processes (AOP) that use oxidation to remove pollutants and membrane technologies, such as reverse osmosis (RO) and ultrafiltration. "As these technologies become more developed and optimized, they are becoming very helpful," she says.

For example, in years past, equipment for water reuse and recycling did not have the capability to achieve the quality of water that is needed for reuse in the process or other areas of the facility in a cost-effective way or within necessary operational parameters, says Joe Tirreno, executive vice president strategic corporate business, with Kurita America (Minneapolis, Minn.; www.kurita.com). "However, in recent years, technologies have advanced enough that wastewater that is discharging can be recycled back into use at the beginning of the plant in a cost-effective manner, especially since the cost of water today is considerably more expensive than the cost of recycling."

FIGURE 1. Water reuse and treatment are intrinsically energy-intensive due to the need to move large streams of water

FIGURE 2. A digital and software-based upgrade often shows processors how to enhance the process intensity or process efficiency to achieve savings in operating expenses while still improving sustainability of the current process

He points to technologies such as ZLD as being improved enough to become a major trend. "We have built several ZLD facilities for the ethanol industry. These plants are often in the middle of a cornfield and do not have access to water or a location to dispose of water, so ZLD is commonly used there. In other industries, ZLD and other technologies are being used for sustainability efforts and for the limited accessibility to raw water."

Often, according to Ravi Chidambaran, chief operating officer with Aquatech International (Canonsburg, Pa.; www.aquatech.com), there are integrated ZLD plants that employ multiple technologies to achieve even higher levels of sustainability. The company recently developed a high-recovery RO technology, Advanced Recovery Reverse Osmosis (ARRO), that overcomes the challenges of conventional RO technologies to achieve recovery rates up to 95% without extended downtime for cleaning and maintenance, which limited previous RO technologies to a 75% recovery rate.

Integrated plants are being built around the world, he notes. For example, in India, Aquatech supplied an integrated ZLD plant that converts waste into pure sodium chloride, which is used as a feedstock in their caustic manufacturing. "Not only do they not incur costs for disposing of the waste, but it's being converted into a raw material with its own value."

The company also developed a technology known as BioMOD AnMBR, which is an anaerobic membrane bioreactor that is an energy-neutral or energy-positive technology, says Chidambaran. "This means it doesn't consume any energy or it can deliver energy that can be used as a heat source. We're able to achieve more than 95% COD [chemical oxygen demand] reduction and generate methane as a by-product, which can be used as a heat source. This also reduces the quantity of extra sludge, so sludge disposal is reduced by almost half."

Alternative treatments

While mechanical equipment options are advancing, there is still a need for additional treatment methods and chemistries, such as environmentally friendly disinfectants, corrosion inhibitors, scale removers or other chemicals, as well as alternative treatment options, such as ultraviolet (UV) light and membrane technologies. So, treatment providers are working to improve these offerings to meet sustainability goals.

One of the simplest options is the use of filtration before mechanical equipment like RO, says Dan Flanick, company manager for Tekleen Automatic Filters (Los Angeles, Calif.; www.tekleen.com). "We offer self-cleaning screen filters with capabilities down to 2 microns," he says. "We've seen a lot of growth for these filters in RO pre-filtration because the membranes for RO are very expensive, so protecting them with a self-cleaning filter saves a lot of downtime and a lot of money on membrane replacement, while helping extend the life of the RO equipment." Prior to the use of self-cleaning screen filters, processors used multi-media filters as a pre-filter, which required a lot of floor space. Tekleen's self-cleaning filters can reduce that footprint by

FIGURE 3. Kurita's DReeM Polymer is a unique chemistry developed to maximize heat-transfer-surface cleanliness and efficiency.

FIGURE 4. Film-forming chemistry, such as SUEZ's E.C.O.Film, is being developed to replace chemicals that contain phosphates, such as those used for cooling water treatment

70%, giving processors additional space to add more equipment at a lower cost, Flanick says.

Resins are another method employed to solve pre-treatment challenges, says Noel Carr, EMEA life sciences and specialties marketing manager with DuPont Water Solutions (Wilmington, Del.; www.dupontwatersolutions.com). "Contaminants are elements, complexes and compounds that are human-made and naturally occurring in water that must be selectively removed so they don't interfere with chemical processing," says Carr. Boron is one of the most troublesome compounds for processors, especially in the microelectronics industry where even a small concentration of boron can create issues for ultrapure water (UPW) systems and product yield. While DuPont has been providing Boron Selective Resins (BSR), most are not applicable for the polishing loop at the UPW system due to the requirements on total organic carbon (TOC) leachables and resistivity. To meet the increasing demand for semiconductor-grade BSR products, DuPont developed the AmberTec UP7530 Semiconductor Grade Boron Selective Ion Exchange Resin, which can be used to remove boron to trace levels with acceptable delta TOC and resistivity. It is ready to use without further post treatment with chemicals at the site, making it an environmentally friendly choice.

And, to protect heat exchange surfaces without harsh chemicals, polymers are being developed for scale prevention. "One of the biggest challenges for chemical processors is the criticality of the heat exchange equipment. It must operate, at the minimum, at design parameters because for every percentage point below design, the impact on productivity and profitability of the facility can be substantial," says Kurita's Tirreno. "This also impacts sustainability because it increases the use of power as the equipment has to work harder, so it's critical to keep this equipment free of scale, but at the same time, environmentally friendly options are needed."

In response, Kurita offers a polymer designed to keep heat exchange surfaces clean and remove existing deposition. Kurita's DReeM Polymer, also referred to as Dispersion and Removal effect Management technology, is a unique chemistry developed to maximize heat-transfer-surface cleanliness and efficiency (Figure 3).

Film-forming chemistry is also being developed to replace chemicals that contain phosphates, such as those used for cooling and boiler water treatments. "Historically, utility chemicals have included some phosphorus component, but we are now seeing bans around phosphors, which create many problems in receiving streams," says SUEZ's Macios. "So, processors are seeking alternative solutions for cooling and boiler water treatment." As a result, SUEZ developed E.C.O.Film for cooling water treatment, which doesn't contain any priority pollutants and is based on simple carbon-, hydrogen- and oxygen-containing molecules that use the natural properties of the water being treated to create an engineered film on the surface of the metallurgy (Figure 4). Specialty chemicals are used to promote the formation of these films on metal surfaces. "We are also using very advanced surface analysis techniques to help ensure that we are creating these films and getting to levels of protection that exceed prior performance of phosphorus materials," says Macios.

Along with kinder chemistries, automation technologies for control and dosing of liquid-solid separation chemicals in wastewater applications are available. Kurita's S.sensing CS system was developed to manage the chemical feed, so the system proactively reacts and adjusts to variations in influent wastewater quality, enabling higher productivity, lower environmental impact and optimized wastewater treatment results. "Typically, in a water treatment facility, you monitor the cleanliness of the water in the effluent and then make adjustments based on that cleanliness, but the time span to see the results between adjustments can be hours," says Tirreno. "This system uses laser technology to measure the influent water to detect production changes, unexpected spills and operational upsets within minutes. S.sensing CS has the capability to make immediate chemical adjustments to changing conditions within the facility, ensuring that chemical costs are lower and water quality needs are met in real time."

Aquatech's Chidambaran agrees: "In general, we see a desire to reduce and optimize chemical consumption and minimize the use of some chemicals that generate potentially hazardous byproducts," he says. "Sometimes, excess chemicals are used continuously based on a programmed worst-case scenario. However, these conditions may only exist for a few days in a year. With real-time sensing, chemical dosing can be drastically reduced or eliminated, based on actual conditions, reducing the chemical cost and also any impact due to unintended byproducts."

Aquatech recently developed Bio-

FIGURE 5. Aquatech's BioFilmPro is a sustainable data-driven solution that effectively controls biofouling by integrating real-time sensing and monitoring of biofilm formation with continuous electrochemical deactivation of bacteria and controlled biocide dosing

FIGURE 6. TrendMiner offers software based on a high-performance analytics engine, which allows processors to search for trends and question process data, enabling them to make data-driven decisions that affect overall plant optimization

FilmPro (Figure 5), a technology solution using real-time monitoring to address such problems. The technology uses an electrochemical method to control biofouling and dose chemicals only when necessary.

Digitalization and analytics

In addition to the available technologies and eco-friendly chemistries offered by water treatment professionals, processors are requesting an enabling technology wrapper surrounding water treatment. "We are not only bringing mechanical equipment, membranes, molecules, chemistries and filters to processors, but we're also bringing sensors, automation and analytics that help them solve problems by having data available around assets so that under any kind of operating conditions, operators can detect, predict and assure performance," explains SUEZ's Macios.

SUEZ's InSight, a cloud-based asset-performance-management tool, uses data analytics to help ensure that water treatment assets operate at optimal performance by connecting key assets, such as boilers, cooling towers, condensers, RO membranes and tanks, into a single platform, which provides real-time visibility at an asset, plant or enterprise-wide level. "This allows users to predict what may be trending in the wrong direction and take action before it becomes a real problem," says Macios.

Kurita's Tirreno agrees that this is of growing importance: "If you have the ability to cognize via sensors and then add the ability to understand the impact via analytics, this results in a step change toward improving water management. Adding the ability to control and make changes remotely, on the fly, such as with our Lumyn technology, allows processors to identify and address potential issues before they happen. Having this interconnection of devices and understanding of how this influences water processes in the plant goes a long way toward improving sustainability."

IntelliFlux's Bhattacharjee adds that managing the data and complexity of water management systems and tying it into the overall health monitoring of the plant helps to make these assets an integral part of the overall process and sustainability effort. "We offer digitalization of the plant through a combination of software technologies and digital twins of the plant, including the water management operations, so that processors can enhance their process intensity or efficiency to achieve savings in operating expenses, enhancements in reliability of equipment, better product quality and improvements in sustainability," he says. "Regarding water treatment, this may include a membrane separation technology with modules that are expensive to replace. Our technology helps reduce the frequency of replacement of those modules, because we can see when they will need to be replaced versus replacing them on a planned maintenance schedule, which provides a cost reduction. Another example might be using the technology to properly monitor chemical dosing of cooling tower water, which reduces chemical usage, thus reducing the cost of chemical management, as well as the waste that is produced as sludge or solids that processors typically have to pay to dispose of."

TrendMiner offers software based on a high-performance analytics engine for data capture in time series, which allows process engineers and operators to search for trends and question process data, enabling them to make data-driven decisions that affect overall plant optimization, including water management, water treatment and sustainability decisions (Figure 6). "We have many different cases where it has been applied to water decisions," says Fischer. "For example, a customer had issues with fouling of the nozzles that pump air through the water to begin a biological treatment process. As soon as the nozzles started fouling, the efficiency of the biological process was reduced. However, when they employed sensors and our analytics, operators no longer had to wait for bubbles in the water to detect fouling. Instead, the sensors detected issues and analytics examined the patterns and amount of airflow to determine when cleaning was needed before fouling occurred, so the process was optimized.

"This ties into energy consumption and sustainability because the nozzles were used to transfer and mix oxygen into the process, but when fouling of the nozzles was prevented via the sensors and analytics, there was a more efficient oxygen supply and mixing operation in the bioreactors, which required less energy consumption," explains Fischer.

Examples such as these demonstrate how digitalization and analytics can improve water treatment and drive sustainability. "As water treatment options continue to advance and improve, it just makes sense to combine them with industrial analytics and IoT [internet of things] to intensify and enhance the efficiency and sustainability of these processes," says Fischer. ■

Joy LePree

Level Measurement & Control

Georg Fischer

Emerson

Pepperl+Fuchs

Endress+Hauser

Measure true level with this sensor and transmitter

The Signet 2260 Ultrasonic Level Transmitter and the 2270 Ultrasonic Level Sensor (photo) provide reliable and maintenance-free measurement and detection for nearly all liquids and types of vessels — from closed-tank systems to open basins. Suitable for use in a variety of water-treatment, chemical-processing and ultrapure-water applications, these ultrasonic devices offer more intelligence than other level-measurement products on the market, says the company. The devices can differentiate between substances like condensation or agitation and the true, actual level of the medium, thereby avoiding incorrect measurements. The transmitter and sensor have accuracies of ±0.05% of the measurement range. Design features include use of a narrow beam that allows safe installation in limited-space tanks and near static objects. The device can recognize foreign matter and disregard them to ensure reliable level measurement, even in tanks where there are vapors, condensation or turbulence. Sensors are available in 4- and 6-m sizes with 1.5- and 2-in. threads. — *Georg Fischer LLC, Irvine, Calif.*

www.georgfischer.com

A radar level transmitter for hygienic applications

The Rosemount 1408H Non-Contacting Level Transmitter (photo) is said to be the world's first IO-Link radar sensor for the food-and-beverage industry. Dedicated features include a hygienic compact design, fast sweep technology and exceptional radar-beam focusing. Non-contacting radar is an ideal level measurement technology for applications that require stringent hygienic facilities and equipment. It is virtually maintenance-free, which helps it to ensure long-term reliability, in sharp contrast to legacy technologies. It has a top-down installation that reduces the risk of product loss through leakage, and it is unaffected by process conditions, such as density, viscosity, temperature and pH. The compact and robust form of the Rosemount 1408H makes it a suitable solution for the small tanks and space-constrained skids commonly used in food and beverage production. The hygienic antenna is flush with the process connection that ensures the removal of process residue during clean-in-place and sterilize-in-place processes, and is insensitive to condensation and build-up. The hygienically approved, IP69-rated device has a stainless-steel housing with minimal crevices to withstand external washdowns and ensure cleanability. — *Emerson, Gothenburg, Sweden*

www.emerson.com

Commission radar devices with HART or Bluetooth

The LCR 10 (photo) and LCR 20 offer continuous, non-contact level and flow measurement (via linearization table), making them suitable for applications in the water and wastewater industry, as well as utilities across all industries. Commissioning is possible via HART, wireless with the company's Level App (application) and Bluetooth on any smartphone or tablet (iOS, Android). The LCR radar sensor is suitable for continuous, non-contact level and flow measurement, especially in storage tanks, containers, open basins and pump lift stations. The hermetically sealed housing allows use in harsh industrial conditions. The device has a measuring range of up to 20 m, and a measurement accuracy of ±2 mm. It operates at process pressures from –1 to 3 bars, and process temperatures from –40 to 80°C. — *Pepperl+Fuchs SE, Mannheim, Germany*

www.pepperl-fuchs.com

Access storage-tank level data from anywhere

Introduced early last year, the Micropilot FWR30 (photo) is said to be the world's first 80GHz wireless IIoT (industrial internet of things) sensor. The continuously recorded measurement data can be accessed at any time and from anywhere via the device's cloud connection. Communication is made possible by an integrated SIM card. Installation is simple and can be done in less than three minutes. This

compact device is suitable for stackable tanks and enables a plug-and-play solution for flexible and easy installation. In addition to the measured level, the user receives information on the location of their storage tanks and containers via local mobile phone masts, enabling users to optimize logistic and storage processes and save valuable time by providing continuous and easy access to inventory information. The free-radiating measuring device covers measuring ranges up to 15 m and temperatures between –20 and 60°C. — *Endress+Hauser, Greenwood, Ind.*
www.us.endress.com

These radar transmitters are easy to configure

Two recent additions to the Sitrans LR100 series of 80 GHz radar transmitters (photo) are compact, and deliver robust, reliable measurements, even in challenging environments. Both deliver fast and easy setup. Sitrans LR140 features 4–20-mA simplicity and is configured via Bluetooth wireless technology and the Sitrans mobile IQ App. Sitrans LR150 offers a four-button user interface on an optional human-machine interface (HMI) for configuration or monitoring. Configuration is also available via Bluetooth wireless technology and the Sitrans mobile IQ App, or remotely with 4–20-mA HART using Simatic PDM. The transmitter becomes operational in minutes using the Quick Start Wizard. — *Siemens Digital Industries, Nuremberg, Germany*
www.siemens.com

Measuring the level in delayed coker units

One of the most critical measurements in a delayed coking unit (DCU; photo) is the level measurement inside the drums. For a long time, neutron backscatter measurements were used for this purpose. In the meantime, this technology is complemented or even completely replaced by continuous gamma level measurement. Among the advantages of this company's nuclear level-measuring devices offer when used in coke drums (photo) are: increased throughput; increased reliability; reduced anti-foam chemical usage; and reduced foam-overs. The challenges of the level measurement, as well as the advantages and disadvantages of these technologies, can be learned in the newest whitepaper from this company. — *Berthold Technologies GmbH & Co. K.G., Bad Wildbad, Germany*
www.berthold.com

This app connects smartphone to level sensors

The Tools app (photo) is mobile-based software that enables the system status to be called up remotely, thereby eliminating the need to climb to the top of a tall vessel, or enter a hazardous zone to read sensor measurements. The sensors deliver data that are not only needed for direct control, but can also provide decisive information for planning maintenance work or eliminating faults. The Tools app offers the ability to remotely operate this company's sensors equipped with Bluetooth, for setting them up, as well as viewing and evaluating their measured values. Current status information can be easily visualized on smartphones and tablets. Now, especially in the case of Vegaswing 60 series level switches, proof testing is said to be easier and more efficient when using the latest version of the Tools app. — *VEGA Grieshaber KG, Schiltach, Germany*
www.vega.com

A new level in capacitance measurement technology

The Capanivo 7000 sensor (photo) is suitable for installations in all kinds of liquids, pastes, foams and slurries. The newly redesigned series now benefits from a new look, new features, a range of new models and the latest IO-Link technology on board. Due to the compact design with 1/2-in. process connection, the sensor is suitable for use in small vessels and pipes. In addition, the sensor, which has been developed in accordance with EHEDG guidelines, meets increased hygiene requirements. In addition to the stainless-steel models, there are also versions made entirely of durable, chemical-resistant plastics, with wetted parts made from PVDF for maximum chemical resistance. With the IO-Link technology, digital data collected by the sensor can be transferred quickly and easily. The CN 7 capacitance sensor works in all types of vessels, pipes and silos. — *UWT GmbH, Betzigau, Germany*
www.uwt.de ■

Gerald Ondrey

Siemens Digital Industries

Berthold Technologies

VEGA Grieshaber

UWT

Safe, Efficient Industrial Lighting

Good lighting at CPI plants is important to prevent accidents. Improved safety can be achieved with LEDs, which are also more efficient and economical than alternatives

Fariyal Khanbabi
Dialight

The chemical industry has long been plagued with safety challenges that make it one of the most hazardous manufacturing sectors of the chemical process industries (CPI). Causing over 18,500 injuries and 25 fatalities yearly on average, accidents in the chemical sector are both tragic and costly, with an annual cost of $676 million [*1*]. Aside from the harsh environment and risk of toxic exposure, the potential for slips, trips and falls alone are a major concern, with fall risk ranking as the number one OSHA (Occupational Health and Safety Admin.) violation for the tenth consecutive year.

While there can be many factors at play in chemical plant safety, poor lighting has been identified as the leading cause of slip, trip and fall accidents and accidents caused by contact with objects and equipment — the second most common types of industrial accidents [*2*]. In fact, upgrading facility lighting to improve illumination and visibility has shown to reduce accidents by as much as 60%. While it may seem obvious that lighting should be a key safety strategy, many organizations simply "make do" with inadequate lighting because it's the status quo.

That had been the case at OQ Chemical's Bishop, Tex. facility — that is, until senior instrument and electrical specialist Marcus Rubio arrived.

"When I came here in 2013 from our Bay City site, I noticed that we had about seven different types of lights in use — everything from metal halides and high-pressure sodium [HPS] to fluorescent, incandescent and even mercury vapor," Rubio says. "That became one of my first goals: to standardize the lighting."

As Rubio found, the inadequate lighting made for poor visibility in many areas. Not only did the lights fail frequently, requiring never-ending and costly routine maintenance, but even when they did work, the light quality itself was poor. Over the last eight years, Rubio has led a massive plant-wide retrofit to modern, industrial-grade light-emitting diode (LED) lighting, and the results have been "a positive for us, all around," he says. "LED is the future of safe, efficient industrial lighting," Rubio confidently asserts.

Why LED leads in safety

As OQ discovered, industrial LEDs are not only more efficient and economical over the long term, but they make for a much safer work environment for employees, which helps lower the risk of accidents and the human and financial costs associated with them. Here's how:

Better visibility. Unlike HPS and other antiquated lighting, industrial-grade LED provides bright, uniform lighting that reduces shadows and eliminates the need for auxiliary lighting like flashlights, which force staff to work one-handed. This improved visibility has proven to enhance trip hazard detection by nearly 24% and help workers identify trip hazards 94% faster [*3*].

Improved color rendering. The dark, orange glow of conventional HPS lighting makes it very difficult for workers to distinguish colors on wiring and safety labels, which can be extremely dangerous in a chemical facility. LED produces a crisp, white light that mimics natural daylight with a much more natural color rendering, allowing staff to easily discern safety-critical colors. "That was another big deciding factor for us," Rubio said. "The light quality has been outstanding, and in seven years, we haven't seen any decline in color rendering like we did with the competition."

Lower fall risk. One of the major issues with conventional lighting is the ongoing maintenance. At OQ, as in many other facilities, that meant crews were often erecting scaffolding and working at dangerous heights just to change a light bulb. Because LED lighting lasts up to three times longer than conventional HPS and similar fixtures, it virtually eliminates the need for staff to work at height to replace bulbs. "We haven't had to relamp a single light in almost seven years," Rubio says of OQ's Bishop facility.

Improved alertness. Especially in facilities with 24/7 operations, worker fatigue can be a serious safety issue. Not only is literally falling asleep on the job a concern, but fatigue can also slow reaction time and reduce alertness, putting sluggish workers at greater risk of an accident [*4*]. LED lighting has shown to improve alertness and reduce fatigue by a factor of five times compared to HPS by suppressing melatonin levels [*5*]. This not only enables workers to think more clearly but also improves their reaction time and efficiency to help them avoid accidents.

Corrosion resistance. Caustic materials — both chemical and salt spray in shoreline locations — can make quick work of conventional fixtures, eroding the fixture housing and causing premature failure. LEDs are much more resistant to corrosive materials with fully sealed fixtures that prevent ingress and corrosion-resistant housings designed to withstand the harsh environment of a chemical facility. This reduces the risk of fixture failure, which not only alleviates the maintenance burden, but also the risk of unexpected poor visibility when a critical light fails.

Hazardous location certified. The presence of potentially flammable airborne contaminants inside a chemical facility presents a huge risk to workers' safety. Even with sealed conventional lighting, the frequent maintenance required forces crews to break the seal often for bulb changes, which risks an explosion. Not to mention, conventional fixtures run extremely hot, which can cause spontaneous combustion of any airborne particulates. LED fixtures not only require less maintenance, therefore lower the risk of explosion, but they also produce much less heat,

FIGURE 1. Good lighting is important to prevent slips, trips and falls

making them safer for potentially combustible environments. A wide variety of models are now available with a range of hazardous location certifications especially for this purpose. "We decided to standardize on Dialight Class I Div 2 fixtures across the plant," Rubio said of OQ. "We didn't want to take a chance of installing a non-hazardous fixture in a hazardous location, so we played it safe and went with all Class I Div 2 for the added peace of mind."

Shock and impact resistance. Conventional filament-style HPS bulbs and fluorescent tubes are notorious for failure and breakage due to their delicate nature. When this happens, it not only rains glass down over personnel, but it can also risk combustion if flammable materials are present. LEDs on the other hand, have no delicate bulbs to break and are much more resistant to vibration, shock and impact. In OQ's welding shop, where pipefitters are often working with long pipe and conduit, the fluorescent fixtures were constantly getting broken. "With the [LED] fixtures, our welding and fabrication team now has much less shadow, better lighting and no worries about breaking glass tubes," Rubio says.

Employee safety is the number one concern in any industrial facility and in the chemicals industry, where there are plenty of environmental hazards to worry about — lighting should never be one of them. As OQ and many others have discovered, upgrading to high-efficiency, long-lasting industrial LED lighting is an essential investment in reducing risk of accidents and injury both immediately and over the long haul. ■

Edited by Gerald Ondrey

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Considerations for Selecting Protective Eyewear

Department Editor: Scott Jenkins

Selecting personal protective equipment (PPE), including safety eyewear, for the chemical process industries (CPI) depends on the hazards present in the facility where it is used. The following describes considerations for selecting eye and face protection based on the potential to injure the eyes or face. Some examples of eye and face hazards and typical sources are summarized in Table 1.

In general, any eye and face protection should be comfortable to wear, sized correctly (if different sizes exist) and should not interfere with the employee's work functions. Poor-fitting eyewear will not afford the necessary protection, may be uncomfortable to wear, and may not be used consistently and effectively.

Impact hazards. Safety spectacles are intended to shield the wearer's eyes from impact hazards, such as flying fragments, objects, large chips and particles. Whenever there is a hazard from flying objects, workers are required to use eye-safety spectacles with side shields. Non-sideshield spectacles are not acceptable for impact hazards. Safety goggles are also intended to shield the wearer's eyes from impact hazards. By fitting the face surrounding the eyes and forming a protective seal around the eyes, goggles prevent objects from entering under or around the goggles.

Face shields. Face shields do not protect employees' eyes from impact hazards when worn alone, and should be used in combination with safety spectacles or goggles for additional protection. Faceshield windows are made of varying types of transparent materials and in varying thicknesses, both of which should be considered when selecting face shields for specific tasks. Window and headgear devices are available in various combinations to be compatible with other PPE, such as hardhats. Welding shields provide eye and face protection from flying particulate matter during welding, thermal cutting and other hot-work activities. These shields are typically equipped with shaded lenses that provide protection from optical radiation.

Heat hazards. Working with heat requires goggles or safety spectacles with special-purpose lenses and side shields. However, many heat-hazard exposures require the use of a face shield in addition to safety spectacles or goggles. When selecting PPE, consider the source and intensity of the heat and the type of splashes that may occur in the workplace.

TABLE 1. HAZARD ASSESSMENT FOR EYE PROTECTION

Hazard type	Examples of hazard	Related tasks
Impact	Flying objects, such as large chips, fragments, particles, sand and dirt	Chipping, grinding, machining, masonry work, wood working, sawing, drilling, chiseling, powered fastening, riveting, sanding and sand-blasting
Heat	Anything emitting extreme heat	Furnace operations, pouring, casting, hot dipping and welding
Chemicals	Splash, fumes, vapors and irritating mists	Furnace operations, pouring, casting, hot dipping and welding
Dust	Harmful dust particles	Woodworking, buffing and generally dusty conditions
Optical radiation	Radiant energy, glare and intense light	Welding, torch-cutting, brazing, soldering and laser work

Source: U.S. Dept. of Labor, Occupational Safety & Health Administration (OSHA)

Chemical hazards. Safety goggles protect the eyes, eye sockets, and the facial area immediately surrounding the eyes from a variety of chemical hazards. The protective seal formed around the eyes is especially important when working with or around liquids that may splash, spray or mist. Several kinds of cover-type goggles are available: direct vented, indirectly vented or non-vented. Vented goggles may be less effective in protecting the eyes from splashes and respiratory aerosols than non-vented or indirectly vented goggles. Face shields are intended to protect the entire face from a variety of chemical hazards, particularly when pouring chemicals or where splashes may occur. Face shields are considered secondary protection and must be used in addition to safety goggles to provide adequate protection.

Dust hazards. Working in a dusty environment can cause eye injuries and presents additional hazards to contact lens wearers. Safety goggles should be worn when dust is present. Safety goggles are the only effective type of eye protection from nuisance dust.

Optical radiation hazards. Welding, thermal cutting, brazing, laser work and similar operations create intense concentrations of heat, as well as ultraviolet, infrared, and reflected-light radiation. Some of these activities can produce optical radiation intensities greater than those experienced when looking directly at the sun. Unprotected exposure may result in eye injuries including retinal burns, cataracts, and permanent blindness. Many lasers produce invisible ultraviolet and other forms of non-ionizing radiation. The Occupational Safety and Health Administration (OSHA) standard 29 CFR 1010.133* includes a table that lists the minimum shade requirements for eye protection during industrial processes that generate optical radiation. The selection of eye protection for lasers should depend on the lasers in use and the operating conditions, and should be consistent with the laser manufacturer's specifications.

PPE and prescription lenses. If employees wear reading glasses with basic magnification, American National Standards Institute (ANSI; www.ansi.org)-approved safety glasses and goggles with reading magnifiers in the lenses are an option. Prescription safety glasses and goggles that are compliant with the ANSI standard are also available, as are ANSI-compliant goggles and safety glasses to fit over prescription glasses. ■

Editor's note: Content for this column comes from the following article: D'Amato, Victor J., Eye-and-Face Personal Protective Equipment, *Chem. Eng.*, February 2009, pp. 48–51.

*www.osha.gov/laws-regs/regulations/standardnumber/1910/1910.133

Production of Vinyl Acetate

By Intratec Solutions

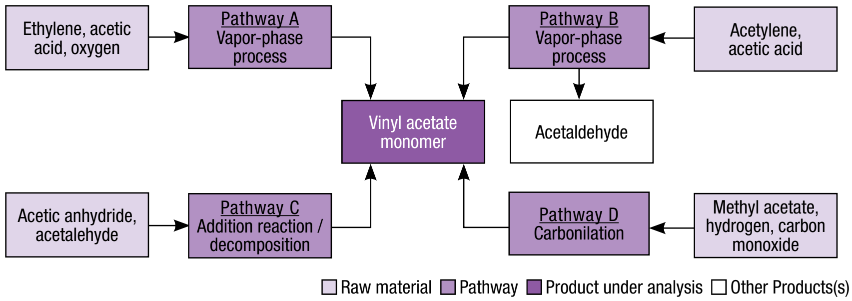

FIGURE 2. Shown here are several pathways for the production of VAM

Vinyl acetate (also known as vinyl acetate monomer; VAM) is important in the manufacture of many polymers and resins. The greatest demand for vinyl acetate is associated with the production of polymers and copolymers, mainly poly(vinyl acetates) used in papermaking, textiles, sealants, binders and coatings; ethylene vinyl acetate, used as a film, in hot-melt coatings and adhesives; as polyvinyl chloride (PVC) alternative; and ethylene-vinyl alcohol, used in food packing and medical applications.

VAM is a colorless liquid with a sweet, fruity smell, soluble in most organic solvents, but only slightly soluble in water. On an industrial scale, the dominant production route of vinyl acetate is based on the reaction of ethylene with acetic acid and oxygen in the gas phase over heterogeneous catalysts that contain palladium.

The main grades of vinyl acetate are technical grade; grade A (99.8%, diphenylamine inhibited); and grade H (99.8%, hydroquinone inhibited).

Production process

The production of VAM from acetic acid and ethylene (Figure 1) comprises three major sections: (1) reaction; (2) separation; and (3) purification.

Reaction. Make-up and recycled acetic acid pass through a vaporizer, along with fresh and recycled ethylene. The feed stream, containing excess of ethylene over acetic acid, is mixed with oxygen, preheated and fed to multi-tube reactors. The reaction occurs over palladium and gold catalysts. Heat is removed by evaporative cooling on the reactor shells. At the end, 8–10 wt.% of ethylene and 15–35 wt.% of acetic acid are converted to vinyl acetate. Water, CO_2 and small quantities of ethyl acetate, ethylidene diacetate and glycol acetates are the main byproducts.

Separation. The reactor effluent is cooled and fed to the pre-dehydration column, where a crude VAM stream is withdrawn from the bottom and stored. The overhead stream is separated into an organic phase, recycled to the column, and an aqueous phase, directed to a decanter downstream. Uncondensed gases are washed by acetic acid. The solution formed is routed to the crude VAM tank, while gases from the scrubber are recycled to the reaction. Part of this gas mixture is washed with water to remove residual acetic acid, and also directed to the crude VAM tank. After water wash, gases are directed to an absorption column, for CO_2 removal by a potassium carbonate solution.

Purification. In the azeotropic column, a VAM-water mixture is distilled and fed to a decanter, along with the aqueous phase separated in the pre-dehydration. Here, an organic phase containing VAM is separated and directed to the light-ends column, while an aqueous phase is routed to the wastewater column, which separates residual VAM from wastewater. Ethyl acetate is withdrawn and discharged, and acetic acid is recycled to the vaporizer. The light ends column strips off acetaldehyde and other volatile compounds from the crude vinyl acetate. Finally, residual acetic acid and heavy-ends are removed in the pure VAM column, yielding a vinyl acetate with 99.9 wt.%.

Production pathways

In addition to the route discussed here (the most common one), VAM may also be produced by the addition of acetic acid to acetylene, addition of acetic anhydride to acetaldehyde, reaction of ethylene with acetic acid and oxygen and the reaction of methyl acetate or dimethyl ether with carbon monoxide and hydrogen. Different pathways for VAM production are presented in Figure 2. ■

Editor's note: The content for this column is developed by Intratec Solutions LLC (Houston; www.intratec.us) and edited by *Chemical Engineering*. The analyses and models presented are based on publicly available and non-confidential information. The content represents the opinions of Intratec only.

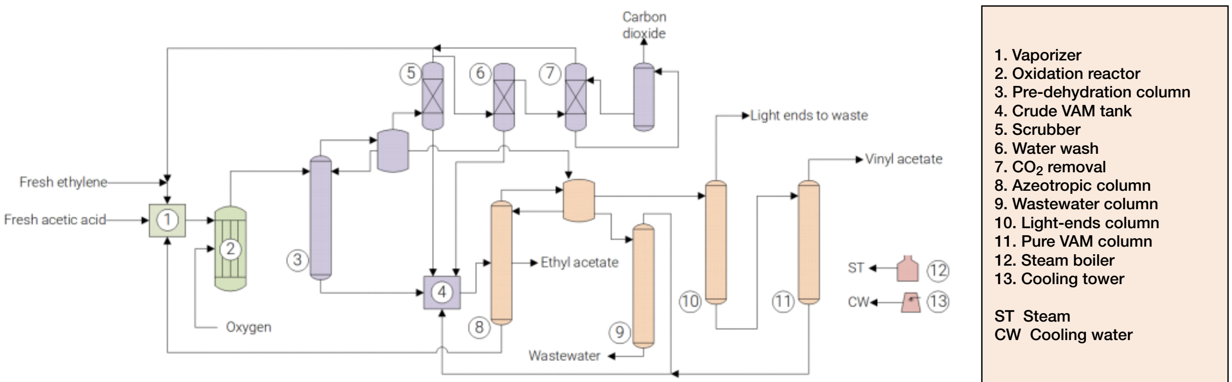

FIGURE 1. The diagram shows the process for manufacturing vinyl acetate monomer from acetic acid and ethylene

Cover Story

Understanding Attrition in Solids Processing

Better qualitative and quantitative understanding of the mechanisms of particle breakage at the level of the particles, the bulk material and the equipment helps minimize the risk, uncertainties and impact of particle attrition in solids processing operations

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The Dow Chemical Company
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Unwanted breakage, degradation or attrition of solid particles during processing and handling is a ubiquitous issue in the chemical, polymer, pharmaceutical, food, mineral, cement, coal, mining and agricultural sectors. While the fundamental mechanisms of attrition phenomena in solids-handling processes are similar to those well researched in comminution (grinding), the varied mechanisms and unquantified state of stresses and strains in solids handling processes and equipment generate additional complexity and uncertainty.

A certain degree of attrition is inevitable during the handling and processing of bulk solids. By applying a fundamental understanding of the physics underlying particle attrition and conducting a systematic analysis of the attrition processes, the negative consequences of attrition can be minimized and mitigated. A two-pronged approach of particle engineering and process engineering must be taken to find the optimal solution. Using the systematic method discussed in this article, the sources of attrition can be identified and mitigated.

Consequences of attrition

In almost all processes involving granules or granular fluid flow, product attrition is a challenge. Among the industry sectors that continue to address attrition are the following: coal, chemicals, petrochemical catalysts, agriculture, cement, detergents, plastics, food, pharmaceuticals and others.

Potential negative impacts resulting from the attrition of solid materials in processes include the following:

- Loss of product due to fines generation — especially when fines cannot be recycled, or the cost of recycling fines is high
- Reduced value due to inferior finish (for example, in food or polymers)
- Loss of activity or functionality (for example, in sorbents, catalysts or seeds and grains)
- Processing problems caused by increased fines (such as segregation, bridging, clogging, feeding, dusting)
- Alterations in end-use applications (for example, fines in polymers, active pharmaceutical ingredient effectiveness in pharmaceuticals, dissolution rates for chemicals)
- Environmental issues (for example, neighborhood impact on process emissions due to fugitive dust)
- Safety hazards of fines, including higher combustibility, explosivity and reactivity, as well as industrial health issues (inhalable dust emissions during processing and end-use)

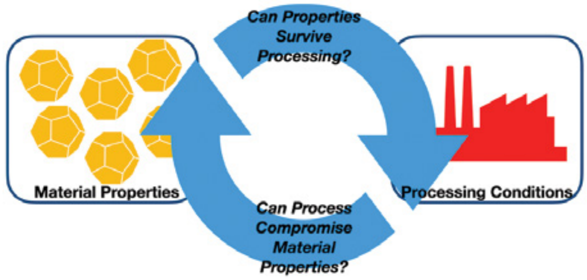

FIGURE 1. The graphic conceptualizes the assessment of material and processing conditions for attrition potential

In most cases, attrition is unintentional and undesirable. Engineers strive to minimize it and its consequences during process design and operation. Attritability (or friability), which refers to the tendency of the particles to break during processing and handling, is not a fundamental property of a material. It is a cumulative (and iterative) consequence of material properties, as well as specific unit-operation and processing conditions (Figure 1). Broad generalizations should not be made about equipment, operating conditions or product families without systematic studies or an attrition audit. Such studies on large-scale equipment and processes are expensive to conduct, and often are not feasible at the design stage.

As with all engineering problems, approaches based on the scientific method (hypothesis generation, testing and validation) are the most efficient and robust. Various tools, such as fishbone plots, affinity plots and "five whys," along with software for statistical analysis, can be used.

To minimize the uncertainties, risks, and impact of attrition on process design, it is imperative to understand (qualitatively and quantitatively) the fundamental mechanisms of breakage at the particle, bulk and equipment levels. The selection of a suitable test method at the particle or bulk level requires an understanding of the mechanisms governing attrition in any unit operation. The challenging part is the quantification of mechanical stresses and corresponding frequencies during pro-

cess residence time. Therefore, for each unit operation in a process, one must delineate the dominant "bulk mechanisms" for particle breakage, their corresponding impact on the type of particle breakage (fragmentation, surface abrasion, chipping, crumbling), and the consequent impact on bulk properties (bulk density, flowability, fines content), which can affect the process step or end use of the product. A holistic view of the process and product engineering is the key to minimizing the deleterious effects of breakage and attrition.

Attrition mechanisms

Attrition occurs when particles in motion come into contact with other particles, or the rigid wall of a vessel or pipe, and the dissipated energy triggers mechanisms for particle breakage. The underlying mechanisms of crack formation, growth and propagation are similar to those observed during comminution or grinding. Much like comminution (grinding or size reduction), attrition is statistical in nature. Not all particles are identical, and neither are the contact or collisions events. One must rely on the measurement of average values or use appropriate statistical functions (such as the Weibull distribution) to quantify it.

As with any solid, failure will occur when stresses exceed the strength of the section to which they are applied. Sharp corners and local aspirates of the small cross-section are vulnerable to modest impact and shear forces that cause an abrasive form of attrition. This vulnerability results in the particles attaining a more rounded form without a significant change of overall particle size, with a degree of fines being created from the broken fragments. More radical fracture of the body of a particle tends to generate a broader range of reduced-size, sharp-edged particles. Attrition is minimized by reducing applied stresses.

Mechanisms at the particle level. The failure modes for a particle can be categorized as brittle, semi-brittle and ductile. The propensity of a particle to fail in a certain mode depends on material properties, strain rate, elastic/plastic state of stress, particle size and contact geometry.

When a particle is subject to external stress (normal or tangential), and the peak stress does not exceed the plastic yield stress (onset of yield), the reversible deformation is considered elastic. Otherwise, the deformation is not reversible, resulting in viscous flow (that is, a dent) or damage to the particle. Any damage to the particle will first be due to brittle failure, resulting from defects, imperfections, microcracks and surface flaws. Brittle failures at high strain rates usually result in cracks that split the particle into fragments. Particles do not fail in a predictable or consistent way because the size and position of the flaws within the particle are varied. The Weibull distribution is commonly used for interpreting fracture data due to brittle failure.

When the impact stress exceeds the onset of yield, plastic flow occurs with viscous energy dissipation. Yet, plastic flow can also generate cracks and microcracks along with compressive radial, normal and tensile stresses during loading and unloading cycles. Radial and normal stress cracks could result in fragmentation, while the lateral stress cracks form chips.

This type of ductile failure can be observed in soft materials where plastic flow ends with a rupture. Plastic flow serves as the precursor to ductile failure. Shear yield stress of the material at the contact via shear chains will determine the extent of damage (ploughing, smearing or cutting). It is known that an oblique angle of attack and shear stress at contact cause damage in ductile materials.

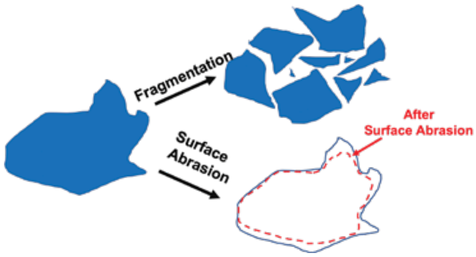

FIGURE 2. The primary mechanisms of particle attrition are fragmentation and surface abrasion

It should be noted that the mode of failure or breakage is not wholly inherent to the property of the material. The strain rate, particle size and shape can change the failure mode. For instance, large particles can fail in brittle mode due to the presence of internal cracks and flaws, along with contact geometry. The fines fraction of the same material can fail in ductile mode due to the absence of flaws and contact geometry. Similarly, high-strain-rate impacts can cause brittle failures, whereas low-strain-rate crushing could result in semi-brittle or ductile failures.

Particle attrition during handling and processing is not a singular phenomenon. The mechanism of particle breakage is broadly categorized as follows: fragmentation/fracture, leading to large sub-fractions; and surface abrasion (chipping), leading to fines (Figure 2). While the breakage manifests itself either as a shift in the peak (the parent peak) of the particle-size distribution or the formation of a new peak in the small particle-size range (daughter peak), a closer ex-

TABLE 1. PRIMARY MECHANISMS OF ATTRITION AT BULK-SOLIDS LEVEL

Mechanism	Description
Bulk shear	Continuous stress during shearing where the load path varies depending on contact renewals; Confinement during shear affects contact stresses
Contact sliding/ friction	When material moves against a boundary, such as a vessel wall or surface of internals, causing sliding, bouncing or rolling motion
Dilated flow	Shearing motion of material with unconfined boundary and/or dilated state allowing sufficient bed expansion during shearing
Impact	Due to rapid change in momentum brought by collision with other particles or walls (such as moving blades)
Crushing	Stressing particles to failure in confinement, either between two rigid surfaces or within a confined bed
Trapping, pinching, chopping	Cleavage or breakage of particles by a moving edge against trapped or confined particle
Vibrations	Oscillatory motion of bed, resulting in frequent low-intensity impacts in dilated bed
Viscous shearing	Shearing action by the fluid medium surrounding the particles

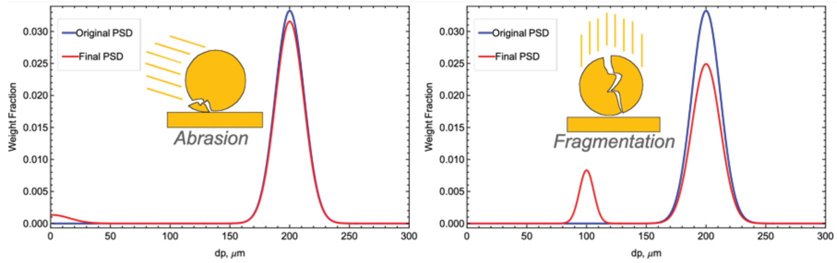

FIGURE 3. Different attrition mechanisms for solid particles show different effects in analyses of particle-size distribution

amination of the particles before and after the handling event can lead to a deeper understanding of the attrition mechanism (Figure 3). Such analysis is necessary to delineate the underlying mechanisms, identify test methods to quantify them, and design and select suitable equipment and operating conditions to mitigate them.

Fragmentation/fracture can occur instantaneously due to the presence of internal or external flaws under subjected stress conditions and loading rates. Multiple repeated impacts (cyclic loading) can cause defects, dislocations or failure planes within the particle, which can subsequently fail due to fatigue. Fragmentation leads to smaller particles of different sizes and shapes that may not resemble the original particle. These smaller fragments may have sharp corners which are then more prone to surface abrasion and chipping.

Fines generated by breakage of asperities due to high compressive stresses, low repetitive tangential stress, and high shear stress at lateral cracks or sharp edges are collectively considered surface abrasion. The overall particle-size distribution largely remains unchanged, except for the formation of a new fines fraction and an increase in particle "roundness" (reduction of edges and asperities).

These two mechanisms mainly occur simultaneously in a system. The relative extent of each mechanism depends on material properties and process conditions. The threshold energy, stress or velocity required to cause fragmentation/fracture is higher than abrasion. As shown in Figure 4, abrasion will occur without fragmentation at low impact velocities, whereas fragmentation will be the dominant mechanism at higher velocities.

Mechanisms at bulk-solids level. Treating the behavior of particles as an ensemble (or bulk) is a practical approach toward developing a mechanistic view of the process. These mechanisms are quantifiable and more amenable to modeling with continuum modeling using average properties. The primary attrition mechanisms at the bulk level are summarized in Table 1.

While the bulk-solids-level mechanisms are well understood for various unit operations, the particle degradation or breakage still occurs at the particle level. Therefore, a fundamental understanding of attrition behavior still requires developing the linkage between bulk-level and particle-level mechanisms. A general guidance for the relationship between bulk- and particle-level mechanisms is shown in Table 2.

Mechanisms at unit operation (equipment) level. Table 3 is an attempt to summarize the contributing mechanisms for particle breakage for key unit operations. The relative impact of these mechanisms on overall breakage will depend on specific material properties and operating conditions. Since typical processes have multiple unit operations, the impact of attrition on all downstream unit operations, and upstream equipment influenced by recycle loops, must be considered.

TABLE 2. RELATIONSHIP BETWEEN BULK- AND PARTICLE-LEVEL ATTRITION MECHANISMS

	Particle-level mechanism	
Bulk-level mechanism	**Fragmentation / fracture**	**Surface abrasion / chipping**
Bulk Sheer	Yes	Yes
Contact sliding / friction	Somewhat	Yes
Dilated flow	Somewhat	Yes
Impact	Yes	Somewhat
Crushing	Yes	Somewhat chipping
Trapping, pinching, chopping	Yes	No
Vibrations	Somewhat	Yes
Viscous shearing	Somewhat	Yes

Attrition testers

An excellent survey of attrition tests can be found in the attrition guide by BMHB [*1*] and in Bemrose and Bridgewater [*2*]. Various standard tests have been documented in ASTM/BS/ISO standards (they are listed in the online version of this article). The list of standards is indicative of the fact that various industries have developed relevant tests to quantify attrition and rank materials for their propensity for breakage (friability).

These tests can be classified as single-particle tests or multi-particle tests (bulk materials). The single-particle tests are suitable for coarser particles (larger than ~300 µm) and measure the integrity of particles through various impact and crushing mechanisms (ASTM D4179). These tests replicate the fragmentation mechanism. For instance, these tests can be useful to compare the strength of coarse particles (such as catalyst pellets) to understand breakage or attrition in packed beds.

Multi-particle or bulk tests are commonly used to compare materials or estimate the extent of fines generation in the process. Commonly used attrition testers [*1,2*] are shown in Table 4.

These testers are typically used for either (i) comparing different materials and their behavior (process function) in a given unit operation (same mechanism, different products) or (ii) comparing different processes to compare attrition rates (different mechanisms at play).

It has been demonstrated in the literature that the relative rankings depend on the specific tester and the testing conditions. Therefore, careful selection of tester and the operating conditions that represent the process of interest is critical for making useful evaluations and comparisons.

Each tester replicates a set of attrition mechanisms and relevant process functions under controlled

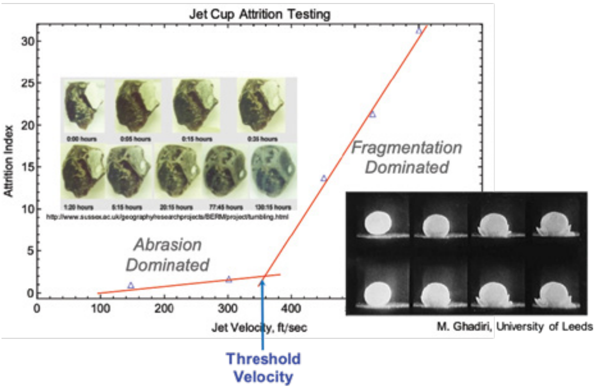

FIGURE 4. The graphs illustrates the concept of threshold velocity. At lower jet velocities, abrasion is more common, while at higher jet velocities, fragmentation dominates

conditions. Therefore, it is critical to match the tester with the inherent mechanism to the process dynamics in question. For example, an impact-based attrition tester is more relevant for dilute-phase pneumatic conveying, whereas a shear cell is more suitable for dense-phase. Thus, before applying any test method, the following items need to be considered:

1. Match key contributing mechanisms between tester and process
2. Choose an appropriate range of operating conditions that match the process conditions. For accelerated tests, make sure that the fundamental mechanism of particle breakage remains the same
3. Agree on the definition of relevant attrition index based on process requirements

Consider the effects of temperature, humidity and fluid properties (such as viscosity and density) on the mode of attrition. For example, certain polymers can fail in brittle mode at room temperatures while in ductile mode at operating temperatures. Similarly, humidity or moisture can affect the strength and ductility of formulated extrudes.

It is important to condition the sample to replicate its condition in the process at steady-state operations. For example, the attrition characteristics of new fluid catalytic cracking (FCC) catalyst can be 5–10 times worse than the equilibrium catalyst (e-Cat).

Quantifying attrition

Particle breakage is generally quantified by comparing the particle size distribution before and after the handling operation. The fraction of particles below a critical particle size can be compared with the original distribution. The definition of critical particle size depends on the unit operation. For fluidized beds, a 20- or 44-µm cutoff is common. This metric is generally only applicable for Geldart Group A powders. For larger particles, the definition of fines will have to be reconsidered. In coke applications (ASTM D4058), particles less than 850 µm are considered fines. Alternately, the ratio of median particle sizes (d_{50}) can be used for comparison. Attrition index is expressed by the following relationship:

Attrition Index (*AI*) =
(%fines < *x* µm at start – %fines < *x* µm after *y* hours) / (%fines < *x* µm at start)

Critical particle size (*x* µm) and test duration (*y* hours) are application- and material-specific.

TABLE 3. MECHANISMS CONTRIBUTING TO PARTICLE BREAKAGE IN SOLIDS PROCESSING UNIT OPERATIONS

Unit operation	Bulk shear	Sliding/ friction	Dilated flow	Impact	Crushing	Trapping, pinching, chopping	Vibration	Viscous shearing
Pneumatic conveying (dilute phase)	-	High	-	High	-	-	-	-
Pneumatic conveying (dense phase)	High	High	-	-	-	-	-	-
Hydraulic conveying	-	-	-	Moderate	-	-	-	Moderate
Silo flow (filling)	-	-	High	High	Moderate	-	-	-
Silo flow (discharge)	High	Moderate	Moderate	-	-	-	-	-
Chutes (full, choked)	Moderate	Moderate	-	-	-	-	-	-
Chutes (fast-flowing)	Moderate	High	High	High	-	-	-	-
Feeder (rotary airlock)	Moderate	-	-	-	-	High	-	-
Feeder (screw)	High	High	High	-	Moderate	High	-	-
Feeder (vibratory)	-	-	High	-	-	-	High	-
Feeder (belt)	Moderate	-	-	-	-	-	-	-
Feeder (apron)	Moderate	High	High	-	-	Moderate	-	-
Discharger (vibratory)	Moderate	Moderate	Moderate	-	-	-	High	-
Discharger (orbital screw)	High	High	-	-	Moderate	High	-	-
Discharger (air blasters)	High	-	-	-	-	-	-	-
Mixer (paddle mix)	High	High	High	Moderate	-	-	-	-
Mixer (plowshare)	High	Moderate	High	Moderate	-	Low	-	-
Mixer (ribbon)	High	High	High	-	-	Low	-	-
Mixer (Nauta mixer)	High	High	High	-	Moderate	-	-	-
Fluidized beds (jet)	Low	-	Moderate	High	-	-	-	Low
Fluidized beds (cyclones)		Moderate	Low	High	-	-	-	-
Circulating fluidized beds (riser)	Low	High	Low	High	-	-	-	Low
Circulating fluidized beds (transfer lines)	Low	High	Low	High	-	-	-	-
Cyclones	-	Moderate	Low	High	-	-	-	-
Screeners (vibratory)	-	Moderate	-	Moderate	-	-	High	-
Screeners (gyratory)	-	High	-	Moderate	-	-	Moderate	-
Stirred vessel (solids only)	High	High	High	-	Moderate	Moderate	-	-
Stirred vessel (multiphase)	High	High	High	-	Moderate	Moderate	-	High

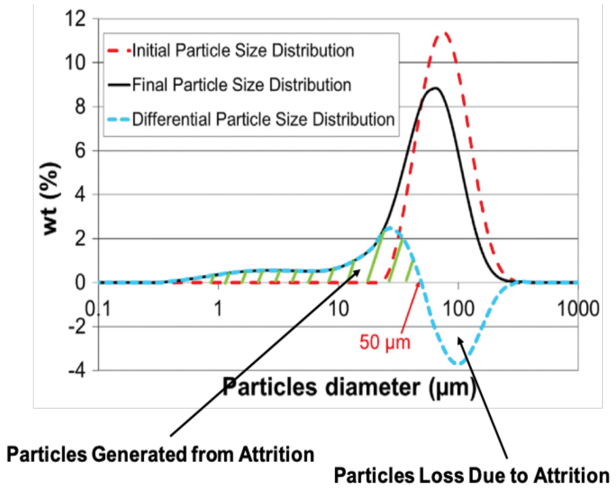

FIGURE 5. The graph depicts the total fines generated index (TFGI), which establishes the relationship between particles generated by attrtion to those lost to attrition

Alternately, AI can be defined as the ratio of median particle sizes when fragmentation mechanism is the dominant one.

$$AI = d_{50,\text{initial}} / d_{50,\text{final}}$$

When considering large particles, attrition indexes based on the generation of fine particles below 20 or 44 µm are insufficient to describe particle breakage mechanisms. Figure 5 shows the particle size distributions (PSD) of the typical attrition test. The red and black curves correspond respectively to the initial and the final particle size distributions. The blue curve corresponds to the difference between the initial and the final particle size distribution. The positive area delimited by the differential blue curve corresponds to the amount of particles generated by attrition, while the negative area corresponds to the amount of particles that were attrited. A new index developed by IFPEN [*4*] is based on the total percentage of particles generated by attrition, which corresponds to the positive area delimited by the differential blue curve shown with the green lines. This index is referred to as the TFGI (total fines generated index) and is shown in this equation:

$$TFGI = M_{generated} / M_{initial} \times 100$$

To obtain reliable particle-size distribution analysis, it is important to obtain a representative sample by following good sampling practices, and select a measurement method that does not cause additional particle breakage during measurement (such as sieving)

When the particles primarily attrit through surface abrasion, particle-size distribution shifts can fall within the band of errors due to sampling and analytical measurement. In such cases, microscopic examination for changes in shape (roundness, edge removal) and qualitative loss of shine may serve as the metrics. However, quantitative measures of shape (shape factors) must be carefully selected with a rational basis. Not all shape factors are equally sensitive to the change in the shape feature of interest.

It is uncommon to use bulk properties, such as bulk density, tap density, shear strength, coefficient of wall friction, for quantifying attrition. However, properties of interest to the end-user should be the final arbitrator. For instance, in grain handling, the fraction of seeds that fail to germinate due to damage during handling is more important than the amount of fines generated. Whatever metric is used, it needs to be applied in the same context, since these metrics are broadly defined in general.

Identifying attrition sources

A systematic approach to understanding and mitigating attrition — which involves looking at particle and bulk behaviors, identifying factors responsible for attrition and developing a hypothesis for action — can solve many attrition-related problems in solids handling processes. The steps needed for this approach are outlined below and diagrammed in Figure 6.

Step 1. Identify key variables in material and process functions. Friability of a material is not a fundamental or state property of the material. That is to say, the path to attrition matters. The potential for, and extent of, breakage during handling and processing can best be understood by analyzing the material function and the process function. Our inability to quantitatively predict breakage from first principles is due to the large number of variables involved and our inability to quantify them within the volume — both temporally and spatially. Often, average values are helpful for empirical correlations, which then require experimental validation or calibration. Key material properties include the following:

- Particle size
- Particle size distribution
- Particle shape
- Density (skeletal and envelope)
- Porosity
- Surface texture or roughness or asperities
- Hardness (crush strength)
- Fracture toughness
- Elasticity/plasticity (brittle/semi-brittle/ductile)
- Young's modulus
- Poisson ratio
- Cracks, microcracks and internal flaws, surface flaws
- Thermal stability
- Chemical stability
- Imaging of particles

Some of these properties are easy to obtain, but others are more difficult. Prioritization is critical with these difficult-to-obtain properties, and needs to relate to the process function. For example, particle size and distribution may be lesser factors for a downward-flowing, inert packed bed than porosity and hardness. On the other hand, particle size and distribution, friability and roughness are more critical in a fluidized bed than hardness. In almost every case, imaging of the material before and after is paramount. Seeing the aftermath of the particle interactions can indicate the mode and sometimes the source of attrition. Key process properties include the following:

1. Particle-particle contact stresses and distribution
2. Internal stresses within particle due to thermal history or chemical exposure
3. Particle-wall stresses and stress distribution
4. Impact angle between particle and walls

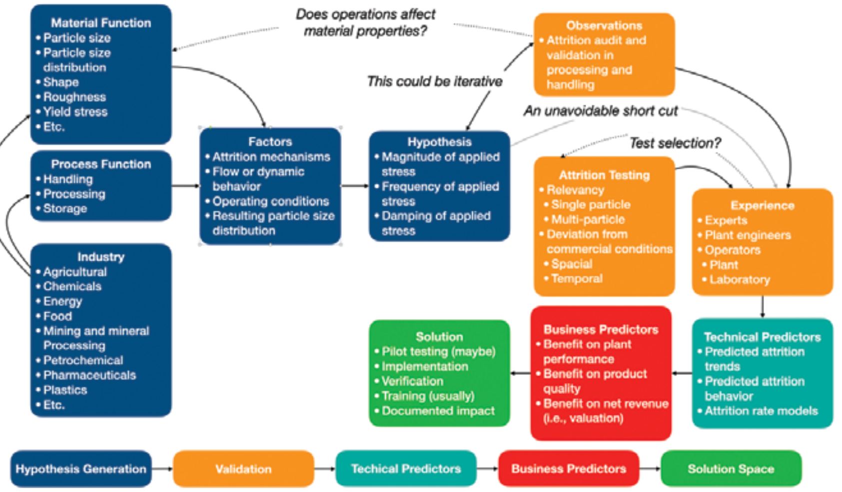

FIGURE 6. The diagram shows a holistic view of the work process involved in understanding and mitigating particle attrition problems

5. Residence time distribution
6. Strain rate (impact versus crushing): particle velocity, velocity gradients and deformation rates of a particle during contacts
7. Confinement during shear and dilation
8. Temperature and temperature gradients (temporal and spatial variations)
9. Wall roughness
10. Ductility or compliance of wall material (vessels, pipes and blades)
11. Particle dispersion (packed bed to dilute suspension)

The system configuration, operating conditions and particle dynamics within a process are responsible for contact stresses, strain rates and failure modes of particles.

Step 2. Rank the factors. The list of potential factors (properties) can be extensive. It is not practical or necessary to measure all the properties to move forward. To narrow down the list to only relevant factors, one must draw upon process knowledge and plant observations. For instance, microscopy or visual dustiness indicates the dominance of an abrasion mechanism, which can be further corroborated by analysis of particle size distribution. Similarly, asperities on the particle are being removed in the processing, which tends to be a lower-kinetic-energy process. If a daughter peak is seen in the particle size distribution, fragmentation is possible, which is a higher-energy process. For fragmentation, the sources of attrition are more limited than for abrasion. If fragmentation appears to not yet be happening, and the level of shear in the process does not seem significant, then perhaps the crush strength could be revealing. One should let the process fundamentals and observations guide the ranking of factors.

Step 3. Generate hypothesis. A hypothesis can be developed with measured material and process properties, and an understanding of the factors that could be responsible for attrition. This is usually an iterative process. Brainstorming tools can be helpful here. There is still guessing at this point, but it is more educated. From the factor exercise in Step 1, one has an idea of the magnitude and frequency of the stress needed. Where would such stresses be in the process, keeping in mind that such stresses are not being dampened? Particles tend to dampen stresses in a flowing system. In regions of dense, slow-moving particles, stresses are dissipated. In regions of dilute, faster-moving particles, a direct and localized impact is likely. Therefore, first look for a source of attrition in the dilute regions for flowing systems.

In non-flowing or slow-moving systems, this is not straightforward. Normal stresses become a factor. That is why the measurement of crush strength is more important for such systems. Crush strengths less than 25 psi suggest that the particle may be too weak for a large packed bed.

The hypothesis needs to focus on the path to attrition and not on the unit operation itself. For example, if the particle-size distribution of the attrited material shows a daughter peak, and imaging shows particles that have been fractured into various fractions, then the hypothesis would be that the attrition is due to a high-kinetic-energy event, such as high-velocity jets, or crushing due to a rotary valve or screw feeder.

Step 4. Validate with observations, experience and testing. The hypothesis provides an idea of what to look for in an attrition audit. Each step of the process should be examined to find regions where the source of attrition is in line with the hypothesis for the cause of the attrition (observations). One would be looking for regions of high shear that could be causing fragmentation, such as a gas jet, screw feeder or paddle mixer where the flight clearances are comparable to the mean particle size or are spinning too fast; a dilute-phase conveying line with abrupt elbows (or too many elbows); or a primary cyclone with a high inlet velocity. Once the potential source(s) of attrition has (have) been identified, it needs to be validated. Consulting experts, plant engineers and operators can validate the source of attrition once identified.

More often than not, attrition problems are not unique. Chances are high that experts have encountered and solved similar problems, and plant engineers, operators or quality-control-laboratory technicians may have suspected the cause of an attrition problem already. These are resources that should be utilized to discuss the hypothesis and observations (experience). Is it in agreement with historical evidence? Do those closest to the problem see the logic in the hypothesis? Was something missed? All of this and more need to be discussed in a group setting.

Even with all parties in agreement, it is value-added to do some local testing to validate the agreed-upon hypothesis (testing). There is a plethora of attrition testing and few standards. Thus, the selection of the attrition test method must be relevant to the hypothesis. Is the velocity or impact used in the testing comparable to plant operations? Can this be done under ambient conditions, or does it need to be done at high temperatures or in a special envi-

TABLE 4. COMPARISON OF ATTRITION TESTERS BASED ON MECHANISMS OF PARTICLE BREAKAGE

Test / operation	Description	Most relevant application	Bulk shear	Sliding/ friction	Dilated flow	Impact	Crush- ing	Trapping, pinching, chopping	Vibra- tion	Viscous shear- ing
Forsythe-Hartwig	Particle bed with single jet	Particle attrition due to jets	Low	-	Moderate	High	-	-	-	Low
Immersed jet (ASTM-D5757	Particle bed with three jets	Particle attrition due to jets	Low	-	Moderate	High	-	-	-	-
Davison cup	Tangential inlet into a cylindrical cup	Particle attrition in cyclones	-	Moderate	Low	High	-	-	-	-
PSRI jet cup	Tangential inlet into a conical cup	Particle attrition in cyclones	Low	Moderate	Low	High	-	-	-	-
Fluidized bed	Pilot-scale fluidized bed with industrial configurations	Particle attrition in cyclones and jets	Low	Moderate	Moderate	High	-	-	-	Low
Jet impingement test	Particle accelerated onto a rigid plate	Particle attrition in cyclones, riser transfer lines and conveying lines	-	High	-	High	-	-	-	-
Particle free-fall impact tester	Particle drop on rigid surface	Particle attrition in feeders	-	-	-	High	-	-	-	-
Drop-shatter tests (ASTM D3038, D4)	Particle drop on rigid surface	Particle attrition due to impact	-	Low	High	High	Moderate	-	-	-
Shear cell	Particle stress induced by shear	Particle attrition in feeders, hoppers, siloes, mixers	High	Low	Moderate	-	-	-	-	-
Tumbler / Rotating drum (baffles)	Baffled or unbaffled rotating drum of particles	Particle attrition due to bulk flow	Low	Low	High	Moderate	-	-	-	-
Ball mill type	Particles are rotated in a drum induced by falling balls	Particle attrition due to bulk flow and crushing	Low	Low	High	High	High	Low	-	-
Vibration / sieving	Particles are vibrated and sieved	Particle attrition in screeners, heat treaters and dryers	-	-	Moderate	-	-	-	High	-
Confined compression	Forced particle compaction	Particle attrition due to compaction	-	-	-	High	High	-	-	-
Viscometers	Impeller rotation in a confined packed bed	Particle attrition in feeders, hydraulic conveying and two-phase systems	Low	-	-	-	-	-	-	High
Paddle wheel	Impeller rotation in a less confined packed bed	Particle attrition in hoppers, mixers and feeders	High	High	High	Moderate	-	-	-	-
Agitated vessels	Asymmetric mixing of impeller in a less confined packed bed	Particle attrition in feeders, mixers and dryers	High	Moderate	High	Moderate	-	Low	-	-
Tabor abrasion tester	Particles confined with imposed shear of rotating plate	Particle attrition in feeders, crushing and contact abrasion	-	High	-	-	-	-	-	-
Single-particle compression test	Particle compression; crush strength of a particle	Particle attrition due to bulk flow, impact, crushing, packed beds, hoppers	-	-	-	-	High	-	-	-

ronment? Is fatigue a factor (suggesting that testing needs to look more at equilibrium conditions than initial conditions)? These questions and more need to be asked and addressed, endeavors typically best done by consulting experts.

Attrition testing further confirms the hypothesis and observations. These test results, coupled with team experience, can narrow down possible causes and provide a path toward solution(s). However, it is critical that attrition testing is representative of the hypothesis. There is a wide range of attrition test methods, and permutations within the methods themselves, so the choice of the attrition test is possibly the most important decision in this work process.

Step 5. Develop technical predictors. Based on the laboratory results and re-evaluations of the hypothesis and re-discussions with your experience team, predictors need to be developed. Predictors are tools that describe attrition trends or behaviors or a mathematical expression to describe the hypothesis. For some processes, a simple trend map is all that is needed. For other processes, a detailed mathematical model is needed. For example, consider a process that has experienced high solids losses due to attrition, and the hypothesis is that the new feedstock from a different vendor may be the issue. Attrition testing confirms that, under the representative testing, the new material is more friable than the older feedstock. Microscopy suggests that the new feedstock is rougher than the old feedstock. The proposed solution may be to not use the new vendor, or to add additional collectors to capture the solids in the process. Economics will decide which is more cost-effective, leading to the Step 6.

Step 6. Develop business predictors. Before a solution is implemented, business drivers need to be developed. These are typically predictors (models, tools) that quantify benefits to the plant in terms of performance, product quality and valuation. Business drivers are quantifiable metrics on what the improvements to the plant will deliver. For example, benefits could include ten months longer reliability for downstream equipment, or a 25% decrease in fines contamination, resulting in an increase in net revenue by 3%. We now have the information to determine the net present value (NPV) or internal rate of return (IRR) for the

proposed modifications to mitigate the attrition issue. If it exceeds the business criteria of the company, then the solution should be implemented, assuming there are no barriers that make it an issue.

Business drivers allow engineers to decide if the solution is value-added. That information will be useful when they need to present a business case. It will also provide the data needed to see if the solution delivers as promised. A technical solution without appropriate business drivers is unlikely to be implemented.

Step 7. Implement solutions. After the hypothesis appears to be valid, as confirmed by testing, experience team and predictors, specific measures to mitigate or eliminate attrition must be proposed. These could include changes in operating discipline, minor changes in existing hardware, replacement of specific unit operations or a totally new process flowsheet. While changes in operating discipline can be easily implemented and tested in the plant, changes related to hardware will require representative pilot-scale testing, which is typically done in collaboration with the equipment vendor. Such testing provides further verification of the drivers and may reduce downtime.

The technical package should consider performance at steady state (lined out) conditions, as well as the effects of upsets and offsets, and should guide recovery with minimal consequences. The comparison of technical options should include capital required, ease of implementation, technical maturity, operability, maintenance, energy and CO_2 footprint, waste and yield. The authors have observed that the complexity and risks of retrofit projects are often under-appreciated.

Even after the solution is implemented and early measurements suggest it is working, the process is not finished. Training may be needed for new equipment or changes with existing equipment. Plant personnel may need training on new work processes. Training is the secret to maintaining all the gains from this work process. Also, documenting how the solutions compare to the business drivers, and noting any lessons learned, is helpful. In order to sustain the gains of the solution(s), it is imperative that a control plan be developed to track and validate the gains.

New processes

All too often, plants involving granular materials are not designed for granular material, including designing with attrition in mind. Designing a new plant where attrition can be a problem is no different from fixing an existing plant with an attrition problem. Instead of finding the source of attrition, one need to target potential sources of attrition and either mitigate or reduce the possible source or engineering around the operation. An example of a new fluidized-bed process illustrates the point.

Fluidized beds are known to have attrition sources associated with the distributors and the cyclones. With access to the material function (properties) and the operating conditions, experts can determine the gas jet velocities, jet penetration length, and cyclone inlet velocity. From jet cup attrition testing, if the catalyst begins to fragment at a threshold velocity of 120 ft/s, then the catalyst is friable and needs to be handled delicately.

First, gas jets can be tempered using shrouds around the jets. Shrouds lower the gas velocity as it penetrates the bed and reduces the number of particles entrained in the gas jet. PSRI has studied the effectiveness of shrouds in fluidized beds and found that a well-designed shroud can reduce attrition by more than four times.

Second is to reduce the gas-inlet velocity in the primary cyclone. Collection efficiency could be compromised, but you can increase the inlet or, better yet, the outlet velocity in the secondary cyclone. Attrition in the secondary cyclone could be high, but less than 5% of the material in the operation is going to the secondary cyclone, so overall attrition is still significantly reduced. If the solids loading is too high for one secondary cyclone because of the reduced efficiency in the primary cyclone, add a second secondary cyclone in parallel. ■

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Effective Contracting for a Pilot Plant

Contracting a pilot plant is a viable approach for any organization. However, it can be fraught with potential issues, which need to be understood and addressed in advance

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IN BRIEF

EXTENT OF THE CONTRACT

LOCATING QUALIFIED CONTRACTORS

TYPE OF CONTRACT

CONTRACTOR LABOR RATES AND PROFIT

IN-HOUSE REQUIREMENTS

MATERIALS AND EQUIPMENT SELECTION

DOCUMENTATION

CONTRACTOR-ORGANIZATION CONTACT

QUALITY ASSURANCE AND CONTROL

Reduced in-house resources have limited the options many organizations have for designing and constructing pilot plants. The increased complexity and intricacy of modern pilot plants also frequently challenge the expertise and resources of many organizations. These factors often force companies to reach outside for assistance in designing and contracting a new pilot plant. In addition to these issues, many organizations have determined that contracting a pilot plant may be the best approach for other reasons. The organization may lack expertise in the area and recognize that it will be cheaper, faster and more effective to have a more experienced pilot-plant firm design and construct the unit. The organization may be overloaded and need to reach out to offload some work (peak shaving). This may be due to a temporary workload increase or as part of a longer-term need the organization is uncertain will continue.

In either case, the expectation is that the need will shortly go away. If the long-term need does continue and the organization feels it will be part of their base load, it may take several years to hire and train the necessary additional in-house staff. These extended needs may also periodically arise again in the future. Some organizations prefer to staff for less than their full normal workload. This allows them to shed staff (the contractors) during downturns and provides significant flexibility without the angst of laying off regular staff. The organization may only have very intermittent needs for a new pilot plant, perhaps only once. In this case, it is more logical to buy the service from outside than to try to learn how to do something in-house that the group may never need again, or at least not for a very long time.

While contracting a pilot plant provides benefits to any organization, there are many potential issues that can arise, which need to be understood and addressed in advance. If they are adequately planned for before the contract, the result will probably be much more successful at a much lower cost. This article describes the common pitfalls and problems that can arise when contracting out a pilot plant, and what organizations can do to avoid or at least ameliorate them.

FIGURE 1. The owner can opt to completely contract out the comprehensive design and construction of the pilot plant, but this option assumes a great deal of trust in the chosen contractor's skills

Note that, when it comes to contractors, most are reputable, professional organizations, but some, sadly, may be less professional and effective than desired, leading to needless delays and cost overruns. While this unsatisfactory group is relatively small, the frustration and lingering problems they create is incredible. Knowing what to look for to avoid such a problematic situation is prudent. Also, understanding the typical challenges that can arise, even when using qualified contractors, can make the project much easier.

Extent of the contract

The first concern is how much of the work will be contracted. The typical approaches

that are usually considered fall into three broad classes, described in the following sections.

Completely contract the design and construction of the pilot plant. In this case, the resulting unit may be installed and started up by in-house resources, or the contractor, as desired. This is the most comprehensive and expensive option. It also has the highest risks, because the effectiveness and success of the pilot plant is inordinately dependent on the contractor's design skill and construction quality, augmented by the thoroughness and quality of the design and construction reviews the organization provides (Figure 1).

Perform the process design in-house and use the contractor to complete the design details. The contractor will handle many details, such as sizing, layout, mechanical design, wiring designs, equipment selection and so on. The pilot plant is normally constructed by the contractor and installed and started up by in-house resources or the contractor as desired. This approach retains the most critical element of the pilot plant — the process design — in-house, thus reducing the risk. However, the cumulative effect of the contractor's design work is substantial and can significantly affect the pilot plant's performance. This approach also requires that the organization be able to supply adequate resources for the required design and to review the contractor details, which may often not be possible.

Develop a fully detailed design in-house, including equipment selection, and hire a contractor to just construct the unit. In this case, unit installation is often included as part of the contracting package. While this requires significantly more in-house resources, particularly design resources, it does ensure that the pilot plant is most closely aligned with expectations. A variant is to contract out the design to a pilot plant specialist and then the construction to a different, usually local, process piping and electrical contractor. This often greatly broadens the range of qualified contractors to do each part of the two-step process, since the construction contractor does not require as high a level of pilot plant experience. It does, however, require locating two qualified contractors and developing two contracts. The extra effort is often ameliorated by the easier time one has in locating qualified contractors.

In all cases, it is not uncommon to contract for an independent detailed design and safety review [*1*, *2*] of the in-house or contract design effort.

FIGURE 2. A contractor that has much experience in large-scale plants may not necessarily bring the correct expertise for a smaller, research-scale pilot plant

Locating qualified contractors

The next concern — perhaps the most critical step — is locating and prequalifying suitable contractors. The most important guideline for successful contracting is to never ask a contractor to bid on a job unless you have prequalified them and feel confident that they can do the work successfully. Prequalification is a tedious and time-consuming process that is often overlooked or reduced to a cursory check by the procurement team to confirm the potential candidate's financial stability and safety record. This lack of prequalification or a cursory prequalification usually results in major problems. All too often, a name is picked to fill out a bid slate without any prequalification. Then, when they submit the lowest or superficially most attractive bid, you are stuck with a contractor that is totally unsuited to the project.

While many factors are involved in prequalifying a contractor, their safety record is of utmost importance. The contractor should be able to answer questions about their safety programs to convey the sense that safety is a priority and not just a buzzword.

While the safety record is very important, it is much harder to find a qualified pilot plant contractor with an outstanding safety record than to locate a safe but unqualified contractor. Only after you find a pilot-plant-capable contractor should you bother looking at their safety record. If their record is poor, it may eliminate them from consideration, but conversely, a great safety record does not a good pilot plant contractor make. Recognize also that smaller contractors may have higher safety indexes, yet be no less safe due to the inherent issues with safety statistics [*3*].

Beyond safety, many other questions must be answered: Is the contractor technically qualified to do the work? Have they done enough similar work in the last few years to be considered experienced? Do they appear knowledgeable on the areas you are interested in? Experienced does not necessarily mean qualified. The contractor may be very experienced in process construction but clueless about pilot plants. They may be able to handle multi-million-dollar projects, but be structured poorly to handle a $100,000 project without overloading it with unnecessary overhead (Figure 2). They may understand wastewater treatment but be ignorant of chemical processes. So, exercise great care in assessing a contractor's experience. Recognize also, that the nature of research often makes it difficult to find anyone who has done exactly what you envision. Hence, you will often have to assess how closely the contractor's recent previous work matches your project requirements.

Other questions to consider relate to areas that are difficult to quantify, but typically easy to recognize. Are they professional? Do they answer calls promptly? Do they provide

FIGURE 3. Obviously, owners want equipment deliveries to be an efficient process that doesn't delay the project, but they should be wary of contractors who provide overly optimistic task schedules, which may not account for certain details or contingencies

suitable information quickly? Does the information make sense? Is it relevant? Do they submit information in an organized and easy-to-understand fashion? Do they understand your questions and concerns or seem to treat them as minor issues or act baffled as to why you are asking them? How do their previous clients view their performance? Would a previous client ask them to bid again? Did a previous client feel they contributed to the success of their project? Did the contracted pilot plant start up easily and operate properly? What lessons did they learn in dealing with the contractor?

Pay attention to body language or verbal clues as you talk to references. Sensing an undertone in the conversation can lead you to keep asking questions that can lead you to a better understanding of the contractor's performance. Answers that seem very bland and cursory may well be indications of a problem the reference is unwilling or unable to discuss, or a reference who is too removed from the project to offer an insight. The fact that the client purchased several pilot plants from the contractor may be a good sign (they are happy with their choice) or it may simply be due to inertia or a lack of viable alternatives. The best approach is to try and find a contact who worked on the project or is operating the unit. Finding that contact may require reaching out to vendors, acquaintances and other contacts if the contractor does not have adequate contact information.

Is the contractor financially stable? Ideally, you don't want your project to be either an inordinate amount of their total volume nor a trivial amount. In the first case, any problems on your project risk them having to respond in a way to ensure their survival. In the latter case, they simply may not pay enough attention to your requirements. Usually, the procurement team can find out this information, but it may be harder if the contractor is privately owned. Being too large or too small a client is not, in itself, a sufficient reason to disqualify a contractor, but it does add another element of risk that needs to be evaluated.

How busy is the contractor? Realistically, most good pilot plant contractors will have a backlog and probably some delay before they can get started on your project. While this may create schedule problems, remember that quality always trumps speed. Recognize that a very busy contractor is likely to be stretched to meet their commitments, and therefore may be forced to use less qualified personnel, which can result in issues.

Be alert for the contractor who promises to meet all your schedule goals no matter how ambitious — it is all too easy for a less professional contractor to suggest later that the extended project delays are simply due to "a better understanding of the project's scope." A good contractor will be very careful about promising too much until a good part of the design is finished, when they understand enough of the details to develop a preliminary schedule. Always ask for a detailed, or as detailed as possible, schedule. Equipment deliveries completed in two weeks are highly suspect, as is uncommonly expedient order processing (Figure 3). A succession of highly optimistic task durations usually points to the probability that cumulative delays due to problems will adversely impact the overall schedule. Vague, broad task descriptions like "piping" or "wiring" leave one very suspicious that their magnitude and duration have been realistically and carefully assessed. And remember, a longer schedule will always involve more overhead and administrative costs, so problems in this area can result in attendant cost overruns down the road.

You should also consider how closely the contractor's initial bid matched a project's final cost. Any contractor should be able to give you these two figures, but when there is a significant divergence, they are usually quick to cite "changes in the client's design" as the key reason. While this may well be true, too often it is because the contractor failed to truly understand the design basis before the initial estimate or failed to understand all the work that would be required and add to the costs. In general, a 2–10% cost growth is often reasonable — more than that can indicate a contractor lacking in estimating or design capabilities. While pilot-plant cost estimating is a very difficult area, and one that is subject to numerous potential problems, one expects that an experienced contractor should be able to get it right the first time [*4*].

At the end of the prequalification process, organizations should eliminate any vendors for which the team has reservations. Even if they are hard-pressed to find a definitive reason, it is much safer to go with someone else or spend the extra time to lay the concern to rest. It is also recommended to be honest with the contractor about any reservations. They may be able to address them or may even agree that they are not the right match.

Type of contract

The next major decision is whether to ask for a lump-sum or a reimbursable (also called a cost-plus or time-and-materials) proposal. These contracts are described in the following sections.

Lump-sum. On the surface, a lump-sum proposal appears to have many

FIGURE 4. The owner must come to an agreement early in the contracting period to avoid overstaffing of the project, which can lead to inefficiencies and spurious costs

advantages. It allows a fast comparison between competing proposals and gives a defined cost and schedule. It puts the onus of doing the work efficiently on the contractor, meaning that the contractor appears to incur most of the risk for unknowns. It allows the owner to take a hands-off attitude towards the work and usually requires the lowest amount of resources from their perspective in following up the project.

Unfortunately, experience has shown that, typically, most of these "advantages" are illusory at best, at least in pilot plants. Any changes — and there will always be changes — are going to be costly.

Any unknowns that result in design revisions or equipment issues will mean more expensive selections. Also adding to the owner's costs are any new information that requires a different process approach, as well as other similar issues endemic to pilot plants. Some of this cost markup is reasonable, including: the need to track, estimate and price the change; develop the design to address the change; and follow up the work to have it done safely and effectively. However, some considerations of the mark-up may not have as much merit — be aware of contractors loading the change with extra overhead, spurious arguments as to the effect of the change, overestimates of the time involved, capitalizing on the fact that the organization has no viable alternative and so on. Sadly, many contractors make their profit from change orders. No matter how well-defined the pilot plant is, changes will be unavoidable. Whether they involve new information requiring a design modification or unexpected corrosion behind a wall, such issues eventually end up adding to the owner's costs. If you think you can transfer all this uncertainty to the contractor, you are wrong. Either they will price the job accordingly with an excessively large contingency, or you will end up in conflict after the event where they may argue that you tried to take advantage of them. In either case, you will pay.

Additionally, in the lump-sum contract, safety becomes solely the contractor's responsibility. While this may sound advantageous on the surface, it actually means that the owner will have little influence over how the contractor addresses it.

FIGURE 5. Due to the lifecycle and operational expectations of a pilot plant, the approach to determining a return on investment for certain pieces of equipment is quite different than for a commercial-scale plant

This can result in a difficult decision as to abrogate the contract due to poor safety performance, with attendant potential legal issues, or having to approve change orders for additional safety measures.

Significant time and effort are wasted on discussions about what is or is not included in the lump-sum contract, what should and should not be included, what is reasonable or unreasonable and a host of similar issues. Such discussions detract from the effort to find the right answer to a problem and often end up causing considerable schedule delays.

Reimbursable (cost-plus). Cost-plus contracts have numerous advantages. The contractor is guaranteed a fixed profit for each hour worked. Change orders are thus no different from any other order. Granted, the work may be costlier if the timing is wrong and things need to be redone, but the costs are easily identifiable with no opportunity for creative estimating, bookkeeping or similar cost-enhancing issues. Safety, productivity and even quality are enhanced because the owner and contractor can determine the best way to handle the tasks that arise.

This approach provides an enhanced ability to parcel out portions of the work to those contractors best equipped to do the work. The general contractor has little incentive to argue to use a lower-cost but less qualified or less competent subcontractor. The organization has a better ability to utilize a known or preferred subcontractor.

The organization can often set up the project so that less defined (or even unknown portions) are included or easily added. Clearly, when defined, these unknowns may add to the cost and schedule, but there are limited or no downsides to adding them at that time.

Also, new approaches can be tried with less risk. Whether they come from the organization, the contractor or other sources, neither side has a position to defend. The new approaches should be trialed — if they work, adopt them, if not, go back to the previous approach. Many effective techniques can result from such trials.

To be fair, there are potential issues with a reimbursable contract approach that need to be acknowledged and addressed up front to ensure success.

There must be an advance agreement (or at least discussion) regarding productivity. If the owner is unhappy, they must have a mechanism in place to request timely changes. This can be as simple as replacing less-productive workers, or as difficult as replacing a less-organized or less-effective supervisor. This often scares owners (who fear they will be straddled with the poorest performers in the contractor's shop) and contractors (who fear that no one will ever work fast enough to suit an owner's unrealistic expectations), especially since determining productivity is fraught with problems and is open to wide and varying interpretations. Typically, neither side wants to keep changing personnel, since they will lose any investment in training and orientation. Usually, it is easy to reach agreement that a personnel problem exists. If not, then a frank discussion between both sides can lead to a reasonable plan to address the problem. The client wants to maintain their reputation, so they have a vested interest in dealing with these types of issues.

Be aware that many organizations have unrealistic expectations as to how long work will take. It is prudent to get an estimate (even on a reimbursable contract) for all the different phases of the work. Often, this will show major disconnects between owner expectations and contractor estimates. If addressed in advance (or at least recognized in advance), this can go a long way toward minimizing problems. Frequently, explaining to the organization all the intervening steps or work details will open their eyes to how unreasonable their initial assessments were. Other times, it may be necessary to find ways to deal with individuals who simply refuse to recognize that good work takes time.

There also needs to be some advance discussion and agreement to prevent over-staffing the project (Figure 4). For example, a large commercial project that has 1,010 people instead of 1,000 is less efficient, but the effect is probably minimal, whereas a small pilot plant project that has six versus four people is grossly inefficient. A frank discussion upfront about how many supervisors will be required is prudent. Similarly, overhead should be discussed early to avoid problems and misunderstandings during the project. The owner should determine how much overhead will normally be charged for timekeeping, materials procurement and similar less-direct functions, as well as how much owner supervision there will be. It may also help the organization to recognize that they have a problem and let them address the issue while they have the most leverage. Where design work is involved, or more complex sequencing, the owner should make it clear that the contractor will need to justify any extra attendees, assistance, or other charges before they are incurred.

Oftentimes, one, two or even five extra personnel may attend meetings to help address issues, but owners should be quick to ask justifiably unnecessary personnel to leave without participating (or incurring charges).

Similarly, the administrative time

required for any change or materials procurement should be fully understood. Sometimes this time is reasonable — for instance, with a rush order that requires several phone calls to locate the part and then several more follow-up calls to get it delivered quickly. Other times are spurious, as in charging administrative hours per materials item when all are placed with the same vendor at the same time. Arguments that someone is employed "essentially full-time" supporting the project may lead owners to ask that contractor personnel be based on site so their need can be evaluated.

There also needs to be some advance agreement about staffing and staff changes. For instance, an owner may assume they were getting a very experienced pilot-plant engineer only to find a new hire assigned. Or, the contractor's top pipefitter or most experienced electrical supervisor suddenly departs to another assignment to be replaced by a less effective performer. The key to addressing such staffing issues is agreement as to the initial staffing for all the lead personnel, such as the project manager, principal designer and site supervisor, before the project starts. Then, there should be agreement that any staffing changes within the contractor's control will be only by agreement. In that case, when the contractor needs a worker elsewhere, the owner can obtain some compensating relief, such as underwriting of extra training or additional orientation.

FIGURE 6. Conducting a detailed design review, either in-house or with an external third-party contractor, is an essential step before construction can begin

Owners should also make sure they fully understand and agree on the rate structure. Overtime rates, supervisor pay, company-provided equipment and numerous other areas can often involve complicated rules and practices. There may still be surprises, but an early agreement can minimize their potential. As well as potentially trust-destroying misunderstandings.

The organization needs to accept that for this approach to reap the most benefits, they will need to be more hands-on and involved. This means devoting time and resources

to the project followup and working with the contractor.

For pilot-plant projects, the cost-plus approach almost always produces a better product at lower cost.

Contractor labor rates and profit

When considering labor costs, recognize that a "lower cost" or a "less expensive" option does not mean cheap. Contractor rates should be carefully examined before commitment. Many contractors, to secure a bid, will underprice the proposal.

In a non-union firm, this often means that the contractor cannot attract enough properly experienced personnel to staff the project because the rates they can pay are too low to attract the best personnel. This leads to problems later, because the contractor is either always short of personnel, resulting in schedule delays and attendant increased overhead costs, or low productivity due to inadequately skilled personnel. In a union shop, where the rates are set, such staffing issues are usually not a problem. Even with higher hourly rates, a more-productive and qualified team may end up as a cheaper option if they complete tasks significantly faster.

This includes not only the trades personnel but also the supervisory and engineering staff. Some smaller pilot-plant contractors hire new graduates or new tradespeople, pay them at the lowest end of the pay scale, and don't expect to retain them long. This results in lower-cost proposals, but no real experience, and almost always costs the organization more in the end. Other pilot-plant contractors have their non-degreed design staff construct most of a smaller unit themselves, arguing that this is more efficient. Usually, either the staff tends to be lower end or lower productivity. Arguments that their having a degree leads to savings as you need less documentation are very dangerous, because these normally translate to less ability to review the product before it is finished. So, determining how the construction will be staffed is important.

Many organizations fixate on contractor profit in cost-plus bids. They feel if the profit is too high, they are paying too much. Realistically, many contractors, particularly smaller pilot-plant contractors, have a very limited understanding of their overheads. As a result, much of what they may call "profit" is really lost in their overhead. They realize they need to charge it to stay in business but either cannot really identify it or do not understand the need to break it out as a separate line item in their proposals. Typical small contractor margins are in the 15 to 30% range, of which half or more can be assumed lost to cover overhead. If the margins are much below 15% "profit," then the contractor is often in financial duress. Therefore, profit generally should not be used as a selection criterion.

In-house requirements

The organization must take the time to define and document all in-house safety, equipment and installation requirements, among others, and should ask themselves a number of focused questions, such as the following examples:

- Is plastic tubing allowed in-house for flammable and toxic materials?
- How close to the relief device setting does the organization feel comfortable operating?
- Do they want block valves or bypasses on all components likely to need maintenance?
- Are there organizational limits on storage quantities, pressure or temperature?
- Which codes does the organization require following?
- Is there a standard piping desired for instrumentation, sampling or other common operations?

This documentation takes time and effort before starting work, which if not performed, leads to needless design revisions and higher costs. A detailed design basis including all this information is prudent, but outside the scope of this article.

Materials and equipment selection

Materials procurement is an issue in all contracted units. Do you specify all the materials or just the critical pieces? A contractor's goals when specifying and ordering materials are usually making sure it does not cost the contractor extra, it is as easy as possible to order, and they can get the materials quickly with as little pre-planning as possible. The organization, however, usually wants to make sure that it receives the right equipment for the envisioned use first, and then worries about the cost and ease of procurement next. If the organization knows what it wants, then it is easy to tell the contractor to base their proposal on that specific equipment. If the organization does not know what it wants, then it either must have the contractor submit information in advance for their approval or trust the contractor's judgement. Either can be a problem. Advance submittals often slow down a project schedule and take a great deal of effort to review. Often, submittals may be rejected before the organization realizes they are not aligned with the contractor on what the equipment needs to do. Even more often, submittals are routinely accepted without any real review, leading to inappropriate selections. Smaller organizations with limited in-house resources may struggle to find the expertise to review submittals.

Most contractors have very limited operational experience (if any). As a result, their ability to understand the effect of lower-cost components on maintenance and operations is often suspect. On the other hand, many components have a long enough life that, in a research world, even their limited life is much longer than required. A plant might be able to argue that paying 50% more for a valve with a 10-year life is economically viable. A pilot plant with a projected three-year life will not see the return on its investment (Figure 5). Conversely, a pilot plant with numerous lower-cost components that fail too quickly will have a terrible downtime record and almost certainly require a much longer and ultimately more expensive schedule to complete the required research program. And every pilot plant requires a large amount of smaller equipment, such as struts, minor instrumentation, gages, valves, fittings, regulators and so on, that, while individually less critical can, in summation, sig-

nificantly affect the pilot plant's operability. Engineers should take notice if the proposed selection has incompatible materials, is operating at the very bottom or top of its range or is not accurate enough.

Documentation

A contracted unit requires even more documentation than normal, since it often uses equipment and components with which the organization is not familiar. The contractor should be required to supply copies of all manuals, purchase orders and similar information on all equipment. The organization needs to spend the time to confirm that these have been provided before signing off on the unit. All too often, the failure of a component during startup or operation leads to a frenzied effort to find a manual that was never provided or a purchase order that is no longer available in the contractor's system.

All drawings of the pilot plant should be in a format the organization can use and should utilize their symbology whenever possible. This is critical to ensure the organization's design reviews are efficient. The final product should include redlined piping and electrical drawings that represent exactly what is being provided and that clearly highlights any differences — even minor ones — from the original drawings. Most differences will be of little or no concern, but some may be critical. For example, in one recent project, the contractor had difficulty in routing some tubing and arbitrarily reduced the height by a few inches to make installation easier. The lower elevation no longer provided the necessary liquid seal and required costly repiping later when the problem emerged.

The organization and the contractor should also have a firm agreement on the mechanism for any changes, even trivial ones, such as switching valve types, changing the order of less-important components and layout modifications. Most are trivial with no impact, and some are subtle but major issues.

Contractor-organization contact

A critical need is to ensure that the contractor has a single point of contact (SPOC) within the research organization. The SPOC needs to be responsible for arranging for any necessary reviews (design, safety, construction and so on). The SPOC needs to be the only one to tell the contractor what to do and the only one the contractor asks about anything. While this is a relatively simple practice, it goes a long way to avoiding miscommunication, misunderstandings and inefficiencies. This puts a large burden on the SPOC and is almost always a full-time job except for on very small projects. Indeed, sometimes the SPOC may need to be a small team (2 to 3 people) to do all the work that is required. Part of the SPOC's responsibilities should include quality assurance and quality control (QA/QC) on the contractor's work as it progresses daily. It is more efficient to inspect work as it progresses rather than to wait for a defined end point. There will be less rework and it will be clear to all contract personnel that the owner is paying attention.

Quality assurance and control

The organization should always ensure that the pilot plant receives a detailed design review either in-house, or by another, independent contractor, before construction (Figure 6). Similarly, the organization must conduct an independent hazard analysis and risk assessment before committing to the final design. Both activities take time and effort and must be factored into the schedule. Also, both activities are likely to result in changes, so holding off on the final cost estimate until these tasks are complete may be prudent. Design-build contracts to a fixed price are rarely viable for pilot plants as there are too many variables in play to produce a valid cost and a high-quality project.

Once construction starts, the organization should arrange for routine frequent visits to the contractor's shop or construction site to review and approve the work. These should be very frequent (weekly is recommended), except perhaps during periods when little is occurring due to deliveries or task sequencing. These visits should be much more detailed than simple tours. Some fittings should be disassembled and confirmed to be properly installed. Tracing of the installed piping should be verified to follow all the drawings. Equipment and instrument installations should be reviewed and confirmed to be acceptable to the organization's standards. Layout issues, always a recurring problem, should be reviewed and resolved. This will take time and effort on the part of the organization. Do not be surprised when this results in many unexpected issues and some change orders. The more design work done upfront and carefully reviewed by the organization, the less these will be a problem. However, some issues will always arise, so adding a small (2–3%) contingency to the final price when the organization gets budget approval is prudent to avoid needless work and resultant delays in seeking approval for small, necessary changes. ■

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Improving a Thermal Loop: The Power Controller

Controlling the temperature of a process using a thermal loop is important in many applications. The importance of a power controller is discussed here

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IN BRIEF

For any process where temperature needs to be monitored or controlled, a thermal loop is required. Typically, a process that contains a thermal loop includes some key elements: the process itself, the temperature sensor or transmitter (often a thermocouple or resistance temperature detector (RTD) plays this role), the temperature controller and the power controller. The goal of the thermal loop is to detect the temperature of the process, and then control it based on certain fluctuations that happen along the line.

The way a thermal loop operates is straightforward. The sensor or transmitter consistently monitor the temperature of the process. As the process fluctuates, the sensor and transmitter will relay this message to the temperature controller (Figure 1). At this point, the temperature controller accepts the input, runs it through its internal algorithm, and sends a signal to the power controller (Figure 2), which in turns affects the process. The best way to explain the functionality of the temperature controller is to think of it as the "brains" of the operation. Alternatively, the sensor or transmitter is the "informant" in the thermal loop. The informant is monitoring the situation, and in its role, informs the temperature controller what is happening in the process. The temperature controller then dictates what needs to be done with the heating or cooling elements in order to eliminate the anomaly.

The next question becomes, "Is there a way to make a thermal-loop control process even more robust and effective?" There certainly is — by adding a feedback function through the power controller.

The feedback

When a temperature controller is informed of the anomaly in the process temperature, it tells the power controller when and how to act in order to normalize it. By its very nature, the controller allows processes to continue running without getting too hot or too cold. This, in and of itself, is a valuable tool. If a process is allowed to continue running at a temperature that is too hot, one risks burning equipment out too quickly or producing an end product that does not meet the requirements set forth. Similarly, if a process is running too cold, the same results could occur depending on the level of tolerance allowed. The mere fact that this is a working model for temperature detection and regulation is positive, but as is often the case, the devil is in the details.

Watlow

FIGURE 1. A variety of temperature limit controllers are shown here

Just as important, if not more so, to the ability of a temperature controller to do its job effectively is being alerted that adjustments are being made. If operators do not know that adjustments are being made due to the detection of temperature anomalies, they cannot adequately prepare for its ramifications.

To illustrate this scenario further, consider the nature of the temperature controller. The functionality of that piece of equipment is to control resistive elements. Often, the objective is to control an entire bank of resistive elements. Let us work from a hypothetical scenario. In this scenario, an operator is trying to control a 10-in. flange heater with 36 elements on it. These elements are wired into three circuits containing 12 elements each. Now, imagine that one of these elements burns out. And then, shortly thereafter, another element pops. As a thermocouple is monitoring the temperature of a process, let us say that it reads through its sensors that the temperature is not up

Watlow

FIGURE 2. Shown here is a power controller

to the required level and must add heat to the process accordingly. Now, let us say that typically the heater can do the heating job for this particular process by operating at 80%. Due to the missing elements, it may now need to operate at 90%. In this case, the temperature controller will adequately compensate for heat loss due to the failing elements. However, there is lag inherent to this process. By the time the temperature is recognized as not being up to the requirement, there may be some product that makes its way through that is not heated adequately. The adjustment will be made eventually so there is not necessarily a colossal problem. However, there is more to this situation than meets the eye.

If there is no feedback in this scenario, the operator has no idea that the heater has lost elements and is now working harder to do the job it was designed to do. Why is this so important? If this is happening, it may be time to prepare for the eventual failure of that heater and get the next one lined up and ready to go. As those in the field can attest, a process heater, depending on availability, can take up to 12 weeks (if not longer) to arrive. Thus, if you know elements are popping, you now have a warning that there are issues with this heater, and you can save yourself the crisis of your critical process shutting down because there is no spare on hand. The amount of revenue that can be lost due to this downtime has decision-makers looking for solutions.

The power controller

Operators can greatly benefit by receiving the type of feedback described above regarding a heater's health by utilizing a power controller. The power controller is inserted into a process in between the temperature controller and the heating or cooling element, cycling the power on and off. There are power controllers available today that have an integrated current transformer (CT), measuring the resistance of the circuit. This means that in the above scenario, where a heater is losing circuits and thus making the heater work harder, a power controller would kick into action immediately. Before the temperature controller even knows that there is an issue with the circuits, the power controller will compensate and route power. This means the power controller can compensate proactively in order to ensure that throughput is not affected. This initiative is not possible with only temperature controllers as discussed above.

In a standard thermal loop, the only way that the temperature controller would know that elements were lost

would be that the process is a bit cooler. It then recognizes that it needs to react to the process being cooler. However, due to the nature of this action, there is an inevitable lag in time, which may result in increased scrap or reduced throughput (whatever is impacted by the process not being quite up to temperature). The power controller solves this problem. As a temperature controller can only react once it realizes the process is cooler than it should be, the power controller, alternatively, notices that the heater is drawing less ohms than usual. Next, additional power can be sufficiently rerouted, so the correct number of ohms is being drawn. Then, through ethernet or a supervisory control and data acquisition (SCADA) system, the loss of elements is communicated effectively to the operator. This proactive approach directly impacts a plant's bottom line in terms of revenue lost otherwise.

Realized benefits

The benefits of implementing a power controller to a process cannot be understated. In practice, there are not many petroleum refineries that have a spare heater sitting on a shelf, waiting to be swapped into a process when the current heater fails. This is very understandable, as some of these heaters can cost in excess of $50,000. Furthermore, without regular monitoring, there is not a general rule to go by in terms of anticipating when a heater may fail. Thus, it is not always fiscally responsible to have such an expensive piece of equipment lying around when the current piece may last for another 10 years or so.

By receiving that precise feedback made possible by the power controller, operators can anticipate far more accurately when a heater may be on its last leg and the investment in a new piece of equipment is both justifiable and required in the immediate future. Then, when the new one is swapped it, the old failing heater can be refurbished.

Another key benefit to the introduction of SCADA-integrated power controllers is the ability to further an automation effort. As decision-makers continue to automate as many functions as possible and scale as many processes as they can, power controllers allow operators to be less susceptible to human error.

These types of power controllers are being introduced into processes in field at an increasing rate. The type of feedback, proactive actions, automation capabilities and ultimate financial benefits that these pieces of equipment can facilitate has decision-makers looking into how they can be integrated into their process. ■

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Perfluorinated Ion-Exchange Membranes: Development and Application

Perfluorinated ion-exchange membranes are developing beyond legacy applications to find use in a wide variety of emerging markets, including renewable energy

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AGC Inc.

Ion-exchange membranes are used in numerous industrial processes, including seawater desalination, electrodialysis and the production of commodity chemicals via chlor-alkali processes. A membrane's materials of construction play an extremely important role in its performance in a given application. For example, ion-exchange membranes based on perfluorinated sulfonic acid (PFSA) polymer show high performance under severe operating conditions. These membranes are available in various grades differing in ion-exchange capacity (IEC) and membrane thickness in order to help end users choose a suitable membrane for their application. Such membranes were originally developed for use in the chlor-alkali industry, but are now used in many kinds of industrial electrolysis applications, such as hydrochloric acid electrolysis and organic-salt electrolysis, and in various electrochemical devices, such as fuel cells and water electrolyzers. In addition, next-generation membranes designed for polymer electrolyte membrane (PEM) water electrolysis are being developed to achieve low membrane resistance, leading to reduced power consumption and lower emissions. In recent years, electrolytic processes and electrochemical devices utilizing separation technology with ion-exchange membranes have been attracting attention in various fields, including renewable energy.

Development led by chlor-alkali

Currently, most PFSA polymer is used in salt form for ion-exchange membranes, which are employed as separators in chlor-alkali cells around the world, where electrolysis splits brine into chlorine and caustic soda accompanied by hydrogen. These basic chemicals are necessary in various industries, including chemicals, textiles, paper and metalworking. Prior to the introduction of membrane-cell electrolysis technology, either asbestos diaphragm-cell or mercury-cell technology had been used in chlor-alkali processes. Environmental issues concerning the use of asbestos and mercury required the development of a safer alternative approach. Although PFSA polymer has excellent chemical stability in the harsh oxidative and alkaline conditions found in chlor-alkali cells, membranes composed of only PFSA polymer lacked the ability to prevent back-migration of hydroxide ions through the membranes, resulting in low current efficiency.

In the 1970s, membranes based on perfluorocarboxylate polymer were developed to reduce this back-migration. The carboxylate polymer enabled membrane-cell electrolysis to be used in the chlor-alkali industry. Through continuous technological advancements, the latest ion-exchange membranes include both perfluorocarboxylate polymer and PFSA polymer in salt form. Low cell voltage or energy consumption has been achieved by the further development of PFSA polymer and other technologies (Figure 1) [*1*, *2*]. Building on the strengths of these established ion-exchange membrane technologies, the industry continues developing new membranes used in a variety of fields.

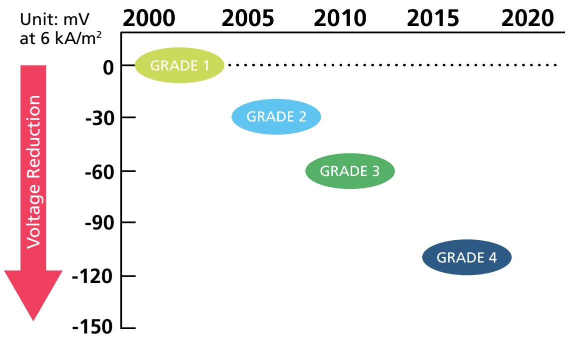

FIGURE 1. In recent years, technological advancements have reduced the cell voltage required for chlor-alkali electrolysis

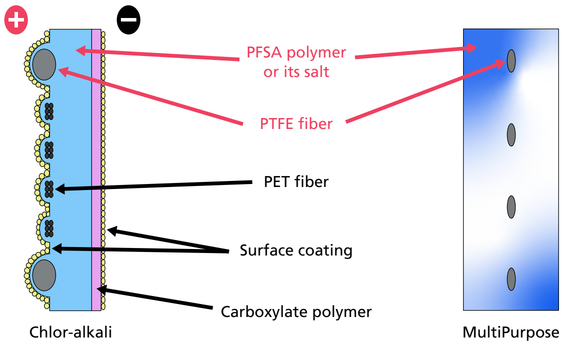

FIGURE 2. The cross-sectional structure of a multipurpose membrane is simpler than membranes used solely for chlor-alkali applications

Structural composition

Generally, the basic structure of an ion-exchange membrane for chlor-alkali electrolysis is divided into three components: an ion-exchange polymer layer, a reinforcement and a surface coating. More specifically, the ion-exchange polymer layer is made through a precursor-lamination process to yield a PFSA polymer in salt form as the low-resistance portion, and a perfluorocarboxylate polymer as the ion-selective portion.

To maintain its mechanical strength and dimensional stability, the chlor-alkali membrane has a mixed woven fabric made of polytetrafluoroethylene (PTFE) yarn and polyethylene terephthal-

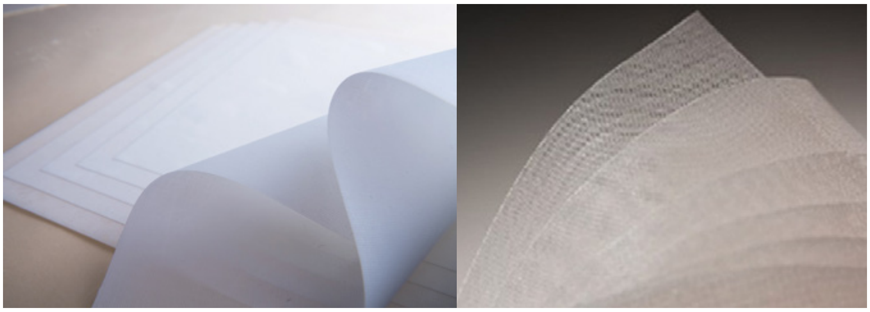

FIGURE 3. A chlor-alkali membrane (left) structure differs from a multipurpose membrane (right)

ate (PET) yarn. The fabric makes it easy to handle the membrane. After starting the electrolytic operation, dissolution of the PET yarn forms cavities in the membrane, leading to a reduction of the cell voltage. Since chlor-alkali electrolysis generates hydrogen gas and chlorine gas in the system, inorganic particles are coated on the surfaces of the membrane to prevent gas adhesion to the surfaces, resulting in further reduction of the voltage [*2*].

Conversely, when compared to specific chlor-alkali membranes, a multipurpose ion-exchange membrane has a simple structure in which PFSA polymer or its salt is used alone for its ion-exchange polymer layer. PTFE fabric is often used as a reinforcement in the membrane. Such fluoropolymer-based materials give the membrane excellent chemical durability. This carboxylate-free composition allows the membrane to perform well, even in a low-pH environment. Figures 2 and 3 illustrate the differences between multipurpose and chlor-alkali membranes.

Advancing membrane technology enables selection of the optimum membrane according to the user's application. There are multiple membrane grades, which differ in IEC and in membrane thickness. High IEC is achieved by introduction of much of the sulfonic acid moiety to the PFSA polymer, resulting in high water content. High IEC and low membrane thickness decrease electric resistance, while low IEC and high thickness increase ion selectivity.

PFSA membranes are produced via an extrusion process using a heat-melted precursor of PFSA polymer, or through a cast process, evaporating solution or dispersion of PFSA polymer. The extrusion process tends to produce thick membranes, while the cast process tends to produce thin membranes. Due to the advancements in both processes, however, the ranges of achievable membrane thickness for the two methods are overlapping. Note that IEC and optimal membrane thickness will vary depending on the end-use application (Figure 4). Membrane performance is also affected by the reinforcement used in the membrane and by the process conditions used during membrane production.

Industrial applications

These simply structured membranes are used as separators in chemical plants and electrochemical devices. An example of such a use is the electrolysis of HCl, which provides the chlorine required for some chlorination processes, such as isocyanates production. Another example is the electrolysis of tetraalkylammonium salts to produce their hydroxides, which are used as the developer to form photoresist patterns in semiconductor manufacturing. They are also used as structure directing agents to synthesize zeolite catalysts.

Other membrane applications include fuel cells and water electrolyzers. Fuel cells are electricity generators whose fuels, such as hydrogen gas and methanol, are oxidized electrochemically to supply electricity. Water electrolyzers (Figure 5) can produce hypochlorous acid as a sterilizing agent and sodium hydroxide as a degreasing agent onsite in many applications, including in food-and-beverage plants and medical facilities [*3*].

Membranes for electrolysis

With the increased interest in renewable energy, power-to-gas technology is drawing more attention in recent years. This technology stores renewable energy, such as solar or wind power (which fluctuate depending on the surrounding environment and weather) in the form of hydrogen or hydrogenated compounds. These compounds can work as energy carriers when needed. An option for converting electric power into hydrogen energy is polymer-electrolyte membrane (PEM) water electrolysis (Figure 6). This electrolysis technology has excellent reaction responsiveness, a wide operating range and the ability to generate hydrogen with high purity and high pressure. It is attracting interest from various manufacturers and research institutes.

Currently, much work is underway to develop new membrane technologies for PEM water electrolysis, as it is emerging as one of the most industrially significant applications based on ion-exchange membrane technology. In PEM water electrolysis, water is supplied to the anode and is oxidized and separated into O_2, protons (H^+) and electrons (e^-). While O_2 is generated on the anode side, the separated proton permeates the ion-exchange membrane and is reduced on the cathode side to generate hydrogen. Thereby, the proton conductivity dictates the energy efficiency of the entire system. In PEM water-electrolysis applications, PFSA ion-exchange membranes can lower electric resistance, save energy and convert renewable energy efficiently. The membrane design requires increasing the IEC

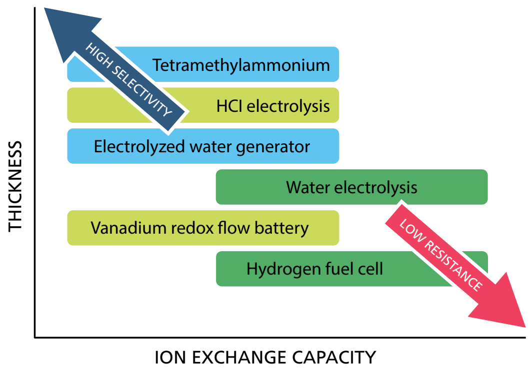

FIGURE 4. Electrochemical performance in various industrial applications depends on the membrane's thickness and ion-exchange capacity

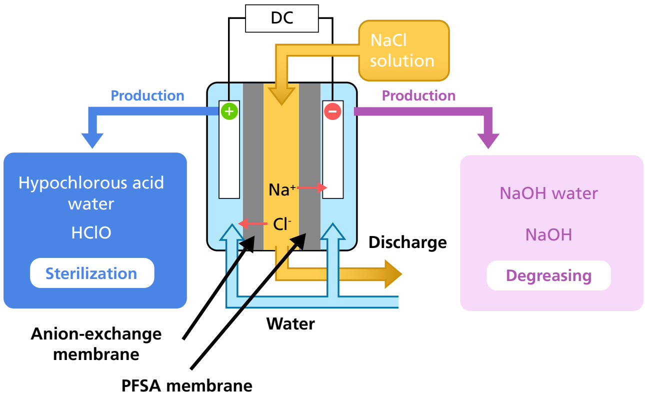

FIGURE 5. A PFSA membrane can be used in a three-chamber electrolyzer for hypochlorous acid and sodium hydroxide

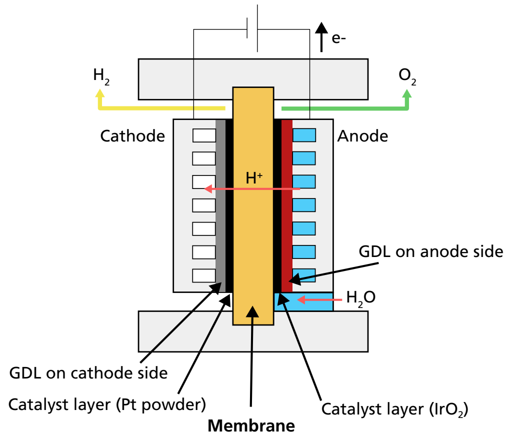

FIGURE 6. PFSA membranes are increasingly finding application as a separator in PEM water electrolysis units

and decreasing the thickness. Both the anode side and cathode sides include a gas diffusion layer (GDL), as shown in Figure 6.

While pursuing low electric resistance, other physical properties, such as mechanical strength and dimensional stability between the dry state and wet state, can be in a trade-off relationship. Therefore, the membrane design should involve optimizing the reinforcement and balancing the thickness. This practice has been learned through decades of previous and ongoing experience in the chlor-alkali industry.

Another emerging application area for ion-exchange membranes is in redox flow batteries, which have been attracting attention as large-scale power-storage devices for renewable energy.

Perfluorinated ion-exchange membranes play an essential role in transitioning the industry to more sustainable operations, in this case, utilizing renewable energy instead of fossil fuels, just as they have done in the past by replacing asbestos and mercury in the chlor-alkali industry. ■

Edited by Mary Page Bailey

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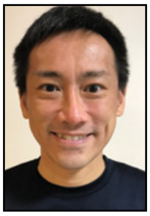

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17. Publication of Statement of Ownership for a Requester Publication is required and will be printed in the November 2021 issue of this publication

18. Signature of Fulfillment Manager: George Severine Date: 9/29/21 PS Form 3526-R, July 2014

Economic Indicators

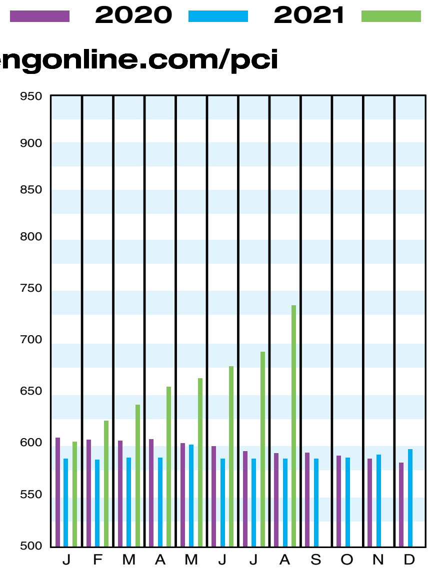
Download the CEPCI two weeks sooner at www.chemengonline.com/pci

CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957–59 = 100)	Aug. '21 Prelim.	July '21 Final	Aug. '20 Final
CE Index	735.8	720.2	594.1
Equipment	919.5	896.8	718.1
Heat exchangers & tanks	787.4	767.5	608.2
Process machinery	921.2	913.4	718.4
Pipe, valves & fittings	1304.7	1245.0	955.3
Process instruments	541.3	531.3	416.9
Pumps & compressors	1148.8	1151.5	1084.0
Electrical equipment	616.8	614.5	563.5
Structural supports & misc.	1000.4	974.8	756.1
Construction labor	347.2	344.0	340.9
Buildings	767.3	765.3	601.7
Engineering & supervision	310.2	310.5	312.1

Starting in April 2007, several data series for labor and compressors were converted to accommodate series IDs discontinued by the U.S. Bureau of Labor Statistics (BLS). Starting in March 2018, the data series for chemical industry special machinery was replaced because the series was discontinued by BLS (see *Chem. Eng.*, April 2018, p. 76–77.)

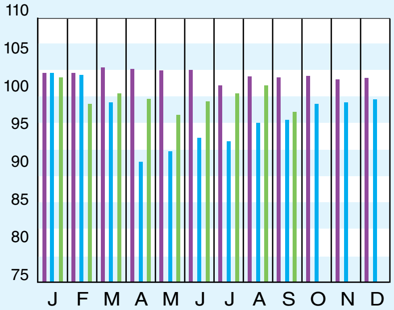
Annual Index:
2013 = 567.3
2014 = 576.1
2015 = 556.8
2016 = 541.7
2017 = 567.5
2018 = 603.1
2019 = 607.5
2020 = 596.2



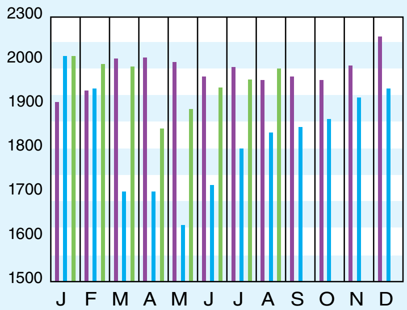
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS		YEAR AGO
CPI output index (2017 = 100)	Sept. '21 = 97.5	Aug. '21 = 98.5	Jul. '21 = 98.3	Sept. '20 = 92.0
CPI value of output, $ billions	Aug. '21 = 1,983.8	Jul. '21 = 1,966.0	Jun. '21 = 1,942.5	Aug. '20 = 1,663.4
CPI operating rate, %	Sept. '21 = 77.8	Aug. '21 = 78.6	Jul. '21 = 78.4	Sept. '20 = 73.1
Producer prices, industrial chemicals (1982 = 100)	Sept. '21 = 345.2	Aug. '21 = 331.0	Jul. '21 = 316.2	Sept. '20 = 225.8
Industrial Production in Manufacturing (2017 =100)*	Sept. '21 = 98.7	Aug. '21 = 99.4	Jul. '21 = 99.8	Sept. '20 = 94.2
Hourly earnings index, chemical & allied products (1992 = 100)	Sept. '21 = 200.6	Aug. '21 = 195.5	Jul. '21 = 195.8	Sept. '20 = 191.8
Productivity index, chemicals & allied products (1992 = 100)	Sept. '21 = 95.0	Aug. '21 = 95.8	Jul. '21 = 355.0	Sept. '20 = 89.4

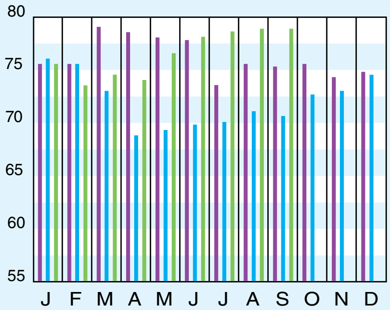
CPI OUTPUT INDEX (2017 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2012 to 2017
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the *CE* Plant Cost Index (CEPCI; top) for August 2021 (most recent available) is once again higher than the previous month, continuing a string of substantial monthly increases since the beginning of the year. In August, the subindices for Equipment, Buildings and Constrution Labor all saw increases, while the Engineering & Supervision subindex saw a very small decline. The current CEPCI value now sits at 23.8% higher than the corresponding value from August 2020. Meanwhile, the Current Business Indicators (middle) show a one-point decrease in the CPI Output Index for September. Also, the CPI Operating rate for September fell slightly, while the producer prices index for industrial chemicals rose.